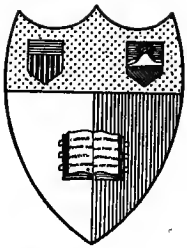
PRACTICAL CHEMISTRY

FOR

ENGINEERING STUDENTS

A. J. HALE



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BY

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WITH AN INTRODUCTORY NOTE BY

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INTRODUCTORY NOTE

One of the practical difficulties encountered by the teacher in carrying out modern schemes of technical education arises from the failure on the part of the students preparing for some particular profession or industry to realise the importance of subjects which they regard as being outside their own province. This difficulty is experienced very generally; it constantly arises, for example, in connection with the professional training of such classes of students as those preparing for medicine or pharmacy, or for the various branches of engineering. It is to this last group of students that the present little work by Mr. Arthur J. Hale especially appeals. Those who are responsible for laying down the broad principles of training for the mechanical engineer have wisely included Chemistry as an essential subject. The leaders of the engineering profession both in this country and abroad are unanimous in urging the importance for engineers of a sound knowledge of at least the general rudiments of chemical science. Such knowledge can only be acquired during the early years of training, and it naturally falls to the duty of the chemical staff in our Technical Schools and Colleges to carry out this part of the students' curriculum.

The successful teaching of a science which, like Chemistry, may at first fail to impress the engineering student as being of any practical utility in his subsequent career, and which therefore arouses no interest unless he has a special aptitude for it, is by no means an easy task in view of the short amount of time which can be allotted to the subject in a two or three years' programme crowded with other collateral

subjects. The degree of success obtainable is of course mainly dependent upon the personality of the teacher—upon his being himself sufficiently acquainted with the requirements of the engineer to enable him to take a comprehensive view of the many points of contact between the two subjects, and so to raise the enthusiasm of the student to at least the point of recognising that Chemistry has a distinct bearing upon his profession.

It is unnecessary to put forward any special plea here on behalf of Chemistry as a subject essential for engineers; its general recognition and its inclusion in the curriculum is sufficient justification for the addition by Mr. Hale of the present laboratory manual to the large number of works on practical Chemistry already in existence. There is, however, one aspect of the question of the chemical training of engineers which is apt to be overlooked, and the present opportunity seems a fitting one for calling attention to the great need in this country of a recognised school of chemical engineering. In all branches of chemical industry useful products are manufactured on a large scale, and the chemical engineer is an essential member of the staff. Chemical engineering is a quite specialised subject, and little or no provision has been made for it in our Technical Schools or Colleges. The engineering education is for the most part mechanical or electrical; but a mechanical or electrical engineering student with a good knowledge of Chemistry is a chemical engineer in the making—he should be more capable of specialising in a neglected field, and should thus be able to give himself better scope for development in a branch of his profession which is not already overcrowded. The young engineer with a sound knowledge of Chemistry is the very man to pass on for specialisation into any school of chemical engineering that may be called into existence. From the same point of view the importance of giving some training to chemical students in the elementary principles

of mechanical engineering has long been recognised in the higher Technical Schools here and abroad. These considerations will, it is hoped, lead to an enhanced appreciation of Chemistry as a subject for engineering students.

The extent to which the scheme of practical work laid down by Mr. Hale can be carried out will obviously depend upon the amount of time that the student can spend in the laboratory, as distinguished from the time he spends in attending lectures. The treatment of the subject in the lecture-room is necessarily more theoretical and descriptive, and the present work, which is essentially for laboratory use, should, under proper guidance from the teacher, be found to be a valuable adjunct to the systematic courses of lectures and tutorial classes which the student is expected to attend during his first and second years.

The programme of practical exercises contained in this little book makes no claim to have introduced any fundamentally new principle; its distinctive feature is the teaching of the subject with a bias towards the use of materials familiar in constructive industry—a bias becoming more and more pronounced as the student progresses, and leading finally to actual specialisation. The principle of teaching science in Technical Schools with a bias towards particular industries, appears to me to be educationally sound, provided specialisation is not introduced at too early a stage. The fundamental principles of chemical science can be developed as philosophically from the study of what may be called "engineering" materials as from those made familiar through the multitudes of existing text-books, and chosen because of the facility with which they can be manipulated by the student so as to bring out the desired general principles. It can certainly be claimed as a matter of experience that such treatment is much more successful in arousing the interest and fixing the attention of the student.

The great danger that the teacher of Chemistry to

engineering students has to encounter is the narrow view held by some engineers concerning the function of that science in relation to their profession. The teacher must never lose sight of the educational as distinguished from the technical value of his subject—of its discipline as a mental equipment quite irrespective of immediate utility. The engineer who narrows his perspective of Chemistry to the analysis of a fuel or of boiler water or flue gas, &c., is virtually asking the teacher to provide him with a man comparable with a workshop apprentice who has acquired manual dexterity in some particular kind of work, but who is devoid of all knowledge of the scientific principles which underlie the construction and use of machinery. The modern teacher of Chemistry will unhesitatingly declare that the *technique* of analysis is in and by itself of no special educational value. Quite ordinary or even inferior students can become skilful in such routine work without having any special aptitude either as engineers or chemists. The engineering profession surely looks for recruits from the ranks of students of wider calibre and whose qualifications are not narrowly circumscribed by manual skill only. For the training of such men the present little manual, rightly used, should be found useful both by teachers and students.

R. MELDOLA.

PREFACE

It is customary for engineering students in our technical schools and colleges, to devote a short period of time to the study of chemistry.

Obviously, such a course is pursued, in order that the student may obtain some knowledge regarding the chemical nature of the materials with which he is particularly concerned.

Such knowledge should be quantitative where possible, and students should be encouraged to analyse those substances which are of prime importance to the engineer.

If he does not, subsequently, during his professional career, conduct the analytical examination of substances which fall in the category of *Engineering Chemistry*, he will find it advantageous to understand, in some degree, the work of the chemist with whom he consults.

In the following scheme, most of the experiments are quantitative, and while inculcating a knowledge of the elementary principles of chemistry, lead directly, and with the least delay, to the analysis of water, fuel, furnace gases, iron, and steel.

The analysis and testing of oils, cements, and alloys is likewise dealt with, while a number of tables containing useful information have been included in the Appendix.

The author desires to express his thanks to Professor

Meldola for advice and encouragement which he has received during the preparation of this work. He is also indebted to Professor Coker, of the mechanical engineering department, for friendly criticism, and to Mr. F. W. Streatfeild, F.I.C., Senior Demonstrator, for many useful suggestions.

Use has been made of many excellent illustrations from various works of reference, which render the preparation of new figures unnecessary, and for the use of which due acknowledgment is made.

A. J. H.

Finsbury Technical College,
London, 1912.

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SCOPE OF THE BOOK

WHILE the programme of work herein outlined is intended primarily for engineering students, it includes a complete course for builders and others who may be pursuing a short course in the *Chemistry of Building Materials.*

The students using this book may be divided into three classes :—

1. Those able to devote three sessions to practical work, each session being made up of thirty-six periods, and each period consisting of two hours.

Such students should work through Chapters I. to VII. in the first session, and in the second session Chapters VIII. to XII.

Chapter XIII. will provide abundant material for the third session.

2. Those able to devote two sessions to the work may omit those experiments in the book which are carried out and fully discussed in the lecture course, and thus complete the programme. Should further deletion prove necessary, they may, in working through Chapters XI., XII., and XIII., confine themselves to those experiments marked with an asterisk.

3. Those able to devote one session only to the work, will find a complete course by carrying out *only* those experiments in the book which are marked with an asterisk. This programme is the one to be followed by students taking a course in the *Chemistry of Building Materials.*

ABBREVIATIONS USED

Ammon. (Am.)	=	Ammonium
Calc.	=	Calcium
Carb.	=	Carbonate
Cm.	=	Centimetre
C.cm.	=	Cubic centimetre
Conc.	=	Concentrated
Exp.	=	Experiment
Gm.	=	Gram
Kgm.	=	Kilogram
Mag.	=	Magnesium
Mang.	=	Manganese
Mm.	=	Millimetre
Potass.	=	Potassium
Ppt.	=	Precipitate
Pptn.	=	Precipitation
Pptd.	=	Precipitated.
Repptd.	=	Reprecipitated
Sod.	=	Sodium
Soln.	=	Solution
Wt.	=	Weight
$\frac{N}{1}$	=	Normal
2N	=	Double-normal
$\frac{N}{10}$	=	Deci-normal
$\frac{N}{100}$	=	Centi-normal

PRACTICAL CHEMISTRY FOR ENGINEERING STUDENTS

INTRODUCTION

WEIGHING—THE BALANCE—GENERAL PRACTICAL METHODS

MUCH of the work in this book is quantitative, and the student is encouraged throughout, to check the quality of his work by weighing the materials used, and the products into which these materials may be converted.

For these "weighings" the balance is made use of, and its importance in chemical work demands a few explanatory remarks concerning the principle on which it works and the manner of using it.

The mechanical principles underlying the construction of the balance cannot be discussed here in full, and the worker is referred to text-books on physics for a detailed description of a delicate balance.

The illustration on p. 2 shows all the visible portions, and it will be noted that there is a beam of rigid but light material, with arms of equal length, which oscillates on a central knife-edge. In using such an instrument for obtaining weights, we really compare the attractive forces of gravitation on two bodies. One of these bodies is a standard weight, and when the attractive force is the same in each case, the beam swings evenly about a position in which it tends to rest. The centre of gravity of the system (pans and beam) is vertically beneath the central knife-edge on which the beam swings; so that, when the bodies in the pans have the same weight, a pointer at right angles to the beam oscillates evenly about the zero mark on the scale.

In order that the knife-edges may not become unduly worn, a rest is provided in the shape of the support *b b*. Upon this, the beam itself normally rests, and not on its knife-edge, while at the same time the knife-edges which support the pans are relieved from pressure.

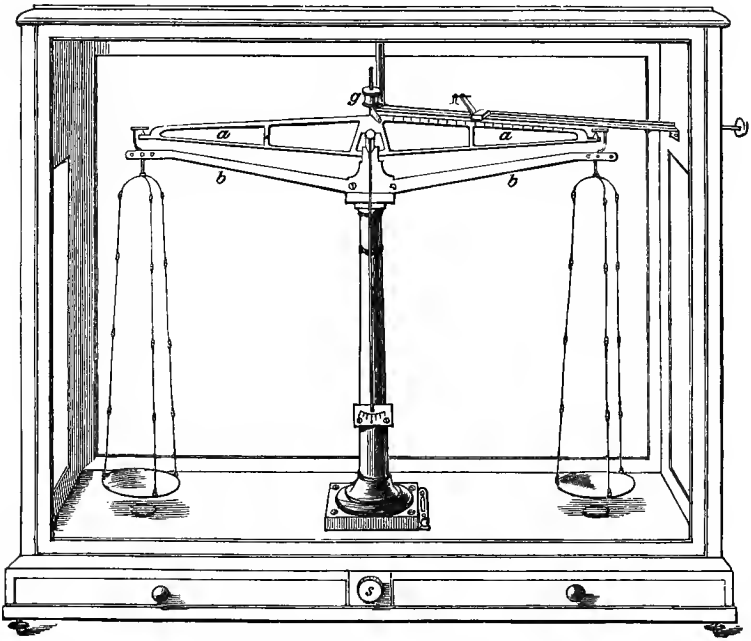

FIG. 1.—(*From Thorpe's "Quantitative Chemical Analysis."*)

a a is the beam of the balance, which consists of an acute rhomboid of light and rigid material.

b b is the horizontal support which is raised and lowered by the lever *s*. When this support is raised it keeps the three knife-edges, *k k k*, just free, by slightly raising the beam and the two scale-pans.

The standard of weight used is the gram, which represents one-thousandth part of the "standard kilogram" of Paris.

The further relation exists, that 1 gram represents the weight of 1 c.cm. of water at 4° centigrade.

This follows from the fact that 1 kilogram of water occupies 1 cubic decimetre, and the relations are :—

1 kilogram of water has a volume of 1 cubic decimetre.

∴ 1000 grams ,, ,, ,, ,,

1 cubic decimetre = 10 × 10 × 10 = 1000 c.cms.

∴ 1 gram of cold water occupies 1 c.cm.

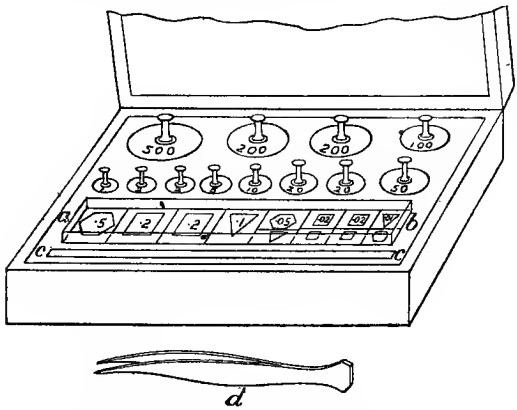

FIG. 2.—(*From Jago's "Inorganic Chemistry." Elementary Science Manuals.*)

This system is called the metric system, because its foundation is the metre, the unit of length.

There are 100 centimetres (cms.) in 1 metre.

The relations between metric and British systems are given on p. 186.

The Weights.—The weights used are kept in special boxes, and each box contains a set.

For most purposes a set reading from 50 gms. down to 0·01 gm. is sufficient.

A set of such weights is shown, *in situ*, in Fig. 2.

The larger weights are made of brass, and are designed so that they may be easily grasped by the forceps.

The weights representing decimal fractions of one gram are made, usually, of aluminium.

Although small weights representing the third decimal figure are included, it is usual to obtain this degree of accuracy with the rider.

This is a light wire stirrup which can be placed at any scale division on the right side of the beam. It is obviously the last weight to be adjusted when a body is weighed, and the number of the scale division on which it rests gives the third decimal figure.

The actual value of each weight is marked upon it (see figure), and each should be replaced after use in its own compartment.

Rules for Weighing.—To facilitate weighing, and to preserve the good qualities of the balance, the following rules should be observed :—

1. Never put anything upon the pans, or remove anything, until the lever is set, so that the beam is at rest.

2. Place the body to be weighed on the left pan and the weights on the right.

3. Put the unit weights in the centre of the pan.
   Put the 1st decimal figure weights on the right side.
   Put the 2nd decimal figure weights on the left side.
   To get the 3rd decimal figure, use the rider on the right side of the beam.

4. The bodies are of equal weight when the pointer oscillates to the same extent on each side of zero.

5. No substance should ever be placed directly on the pan, but should rest on a watch-glass or other suitable receptacle.

6. Form a habit of placing the weight-box as near as possible to the right-hand scale-pan, so that you may always use the right hand for manipulation of weights, while the left hand rests on the lever-screw, serving to raise and lower the beam.

7. Always pick up the weights with the forceps provided, *not with the fingers.*

8. Before replacing the weights in the box, always count up the value of the empty spaces. This affords a ready means of checking the value obtained by reading the weights on the pan.

Measuring.—The preceding statements explain that the unit of volume in the metric system is the cubic centimetre, or, for large quantities of fluid, the litre, which equals 1000 c.cms.

The following measuring vessels are used in chemical work, and they are graduated to contain the correct volumes at ordinary room temperature, 15·5° C.

The Graduated Cylinder.—A cylinder of glass, with graduations marked on it, so that the volume of liquid inside may be read off.

These are made generally in sizes of 100 c.cms., 250 c.cms., 500 c.cms., or ½ litre, and 1000 c.cms., or 1 litre.

These cylinders have intermediate graduations, so that the above figures only represent their maximum capacity. They are made to contain the volumes of liquid represented by their markings.

The Burette.—This is a glass tube of about 1½ cms. bore, graduated in cubic centimetres and tenths, and possessing a maximum capacity of 50 c.cms. It is used for *delivering* a measured volume of liquid by opening the tap or clip which closes the lower end. The figure shows one burette clamped in a burette stand.

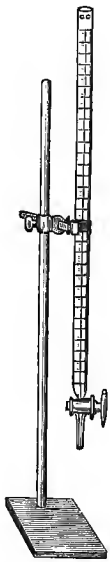

FIG. 3.—(*From Newth's "Elementary Practical Chemistry."*)

The Pipette.—This is a glass vessel used for delivering an exact volume of liquid. It has only one graduation mark on it, and consequently is only used for delivering exactly the volume of liquid mentioned on the instrument.

Pipettes are made to deliver 5 c.cms., 10 c.cms., 15 c.cms., 20 c.cms., 25 c.cms., 50 c.cms., and 100 c.cms.

The liquid is drawn above the mark by sucking at the top end and then quickly covering the opening with the finger. Drops are then allowed to fall until the mark is just reached.

The Measuring Flask.—These flasks are made to hold 100, 250, 500, and 1000 c.cms. exactly, and are *not* used for delivering at all. A graduation mark on the neck indicates the level to which the liquid should rise in order

to give the volume inscribed on the flask. The liquid in these flasks is shaken by holding the stopper firmly and inverting two or three times.

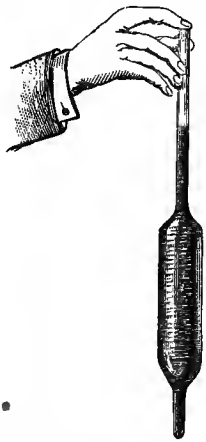

FIG. 4.—(*From Newth's "Elementary Practical Chemistry."*)

FIG. 5.—(*From Newth's "Manual of Chemical Analysis."*)

Note.—In reading water levels in graduated vessels, the lowest part of the meniscus must be noted.

GENERAL PRACTICAL METHODS

Solution.—When two or more substances by mixing, yield a completely homogeneous substance (identical in all parts), the mixture is termed a solution.

Solutions may be solid, liquid, or gaseous.

More generally, the expression is applied to the case of solids mixed with liquids. In these cases the liquid is present in large excess, and the solid becomes completely dissolved and disappears from sight. The resultant liquid is clear and transparent in all parts, and no evidence of the existence of the solid is apparent unless it colours the solution. Examples of solution are salt or sugar dissolved in water.

Evaporation.—Evaporation takes place at the surface of a liquid. By passing into the atmosphere as vapour, the liquid volume becomes less and less, and may ultimately disappear.

Any solid which was dissolved in the liquid remains behind, forming a residue.

The process of evaporation is accelerated both by rise of temperature and by increasing the surface exposed to the air. For this reason a liquid is always evaporated in a shallow dish which stands on a sand-bath, a wire gauze, or a hot-water bath.

Crystallisation.—Crystallisation takes place when a clear solution of a substance is evaporated, so as to drive off excess of the solvent. By the removal of solvent, the solution ultimately becomes "saturated," and on cooling such a saturated solution most of the dissolved solid crystallises out. The rule to be observed when crystallising a clear solution is: evaporate by heating, until crystals begin to form on the surface of the hot liquid; then remove from the source of heat and allow to cool. When quite cold drain off the clear mother-liquor, and dry the crystals by placing them on a filter-pad or porous plate.

Precipitation.—Frequently, when two clear solutions are mixed together, a separation of solid matter takes place. The resultant solid, which gradually falls to the bottom of the liquid, is spoken of as a precipitate, and the process as precipitation.

Decantation and Filtration.—When a solid in suspension, *i.e.* a precipitate, settles to the bottom of the liquid readily, it may be separated from the liquid by pouring off, *i.e.* decanting, the latter. The solid remains behind at the bottom of the vessel.

Filtration is a more complete method of separating a precipitate (ppt.) from the liquid in which it is suspended. The mixture is poured on to a filter-paper fitted in a funnel, and the liquid runs through, while the solid remains behind on the filter-paper.

To fit a filter, the circular filter-paper is folded to form a semicircle. This is then folded again to form a quadrant, and by opening the paper, folded in this way, a hollow cone is obtained which can be fitted into a glass funnel. When

filtering a liquid, the funnel must be placed in a filter-stand (Fig. 6). The liquid must never reach quite to the top of the filter-paper, and one lot should run right through before fresh liquid is added, especially when the work is *quantitative*. The liquid must be poured in carefully, not "splashed" in, and when the work is *quantitative* the pouring must be done by the aid of a glass rod, to prevent splashing and also to prevent drops of liquid and

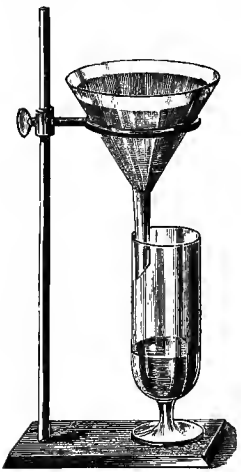

FIG. 6. — (*From Newth's "Elementary Practical Chemistry."*)

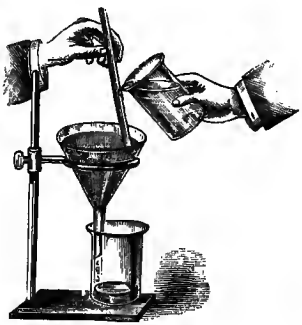

FIG. 7.—(*From Newth's "Elementary Practical Chemistry."*)

precipitate from running down the side of the beaker (see Fig. 7). If the ppt. on the filter-paper is to be washed, this is accomplished by a well-directed stream of distilled water from a wash-bottle (see p. 12). All ppts. should be filtered while hot, and washed with hot water unless otherwise directed. This accelerates the rate of flow through the funnel, and this end is also reached by always making the leg of the funnel touch the side of the receiving vessel, as indicated in Fig. 6. Any splashing is also prevented by such an arrangement.

Drying.—To dry a ppt. in a filter, place it (while in the funnel) in the hot-water oven, after allowing to drain. A quicker method is to place the filter in a hot-air oven at 110° C.

A third method for quick-drying is to place the filter and funnel in a tin cone, which rests on a wire gauze (Fig. 8). By placing a small flame underneath, the filter is soon dried completely.

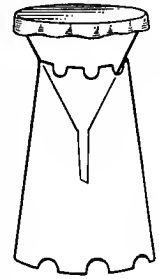

FIG. 8.

Crystals may be dried by first well draining, and then placing on a filter-pad or between two sheets of filter-paper. If they are well drained it is seldom necessary to place them in the hot oven, and a final drying may be accomplished by well pressing between dry filter-papers.

Desiccation.—Desiccation is a process of drying a substance or keeping it in a dry state, by placing it in a vessel which contains some material having a great affinity for water vapour. Sulphuric acid and calcium chloride are such materials, and they are spoken of as desiccating agents. The vessel used is called a desiccator (Fig. 9). The dryer is placed at the bottom of the vessel, and the substance to be desiccated rests on a shelf in the centre of the desiccator. The joint between the lid and the vessel is made air-tight by greasing with vaseline.

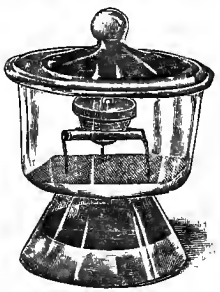

FIG. 9.—(*From "Newth's Manual of Chemical Analysis."*)

The Bunsen Burner.—This form of lamp is used in all chemical experiments which require heating on a small scale. The air-hole at the base can be varied in size, and by this means the flame may be made luminous or non-luminous. The latter is the hotter flame, and is used for all strong heating; it possesses the further advantage that it deposits no soot. This flame is obtained when the maximum amount of air mixes with the gas. As the air-inlet is made smaller, so the flame becomes more luminous, and when the air is shut off com-

pletely an ordinary luminous coal-gas flame is obtained. With the air-hole open, the flame has a 3-cone structure. The outer mantle is the zone of complete combustion and the source of heat, the hottest portion being in the region A. The inner cone, B, consists of unburnt gas, and is comparatively cool (Fig. 10). It is surrounded by the blue cone, D. The outer mantle has an oxidising action since it is mixed with air in excess, and small objects like borax beads and

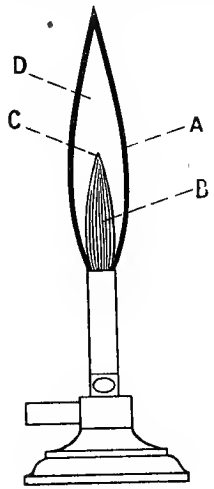

FIG. 10.

FIG. 11.—(*From Newth's "Elementary Practical Chemistry."*)

flame colouration wires should be held near A, the hottest part of the flame. The reducing flame is obtained by shutting off the air slightly, when a luminous point makes itself visible at C. This small luminous area is suitable for reducing borax beads.

Blowpipe Flame.—The blowpipe flame is obtained by blowing into a small luminous flame. The position occupied by the nozzle of the blowpipe and the relative size of the flame obtained is shown in the figure, where the manner of heating a substance on charcoal is also shown. A good blast of air in the position shown gives a hot oxidising flame.

In order to get a luminous reducing-tip, the nozzle must be held a little way from the burner, and the air-blast must not be so strong.

To Bend Glass Tubing.—For this purpose an ordinary luminous "fish-tail" burner is used. The glass tube is held by the ends in the manner indicated in Fig. 12, and continually rotated. As soon as softening commences, remove the tube from the flame and bend it to the desired angle. Sharp ends of glass tubing or rod may be rounded, by rotating them in the Bunsen flame.

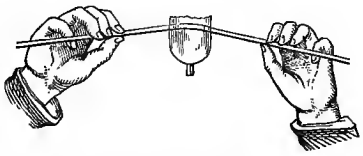

FIG. 12.—(*From Thorpe's "Quantitative Chemical Analysis."*)

Ignition Tubes.—These can be made by selecting a piece of glass tubing 15 cms. long, and having a diameter of 0·7 to 0·8 cm. The ends should be rounded, and then the tube rotated with its centre in a Bunsen flame. When the glass is thoroughly soft, pull it out quickly and allow to cool slightly. By pressure with a file the fine ends are broken off short; the end is sealed by rotation in the flame, and, while hot and soft, the thickened portion is blown out by applying the mouth to the open end. In this way two ignition-tubes will be produced. (See Fig. 13.)

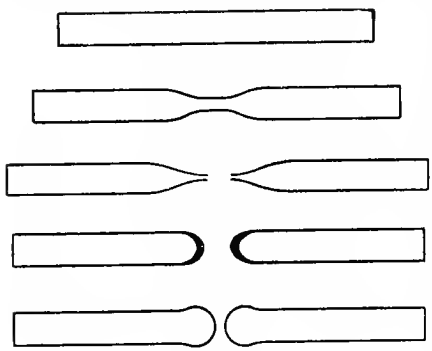

FIG. 13.

Cork Boring.—Holes are bored through corks by means of a cork-borer. The edge must be thoroughly sharp before boring is started, and then the rotation of a sharp edge is

to be relied on rather than pressure. Excessive pressure always leads to a tearing of the cork, especially if the borer be blunt.

For boring holes in ordinary corks, a borer must be selected which has a diameter slightly less than that of the glass tubing used.

For rubber corks, a borer whose diameter is slightly larger than the glass tubing must be taken. To facilitate the turning of the borer in rubber, it may be moistened with water or methylated spirit containing a little soda.

All ordinary corks must be softened before boring, by rolling them on the floor, with the foot.

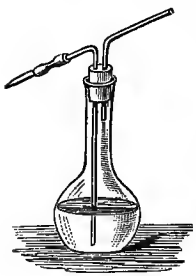

FIG. 14.—(*From Thorpe's "Quantitative Chemical Analysis."*)

The Wash-bottle (see Fig. 14).—The capacity of the flask should be about $\frac{1}{2}$-litre. A cork is selected, softened, and then two holes bored side by side. The figure indicates the relative length of each tube, and also the angle most suitable. To the end of the acute-angled tube a nozzle is attached by rubber tubing. The nozzle can be made by drawing out a piece of glass tube of the right diameter. All ends should be rounded by rotation in the Bunsen flame before finally fitting the parts together. By blowing down the short tube a fine jet of water can be directed from the nozzle.

In order to facilitate the holding of the washing-bottle, when it contains hot or boiling water, a wrapping of twine or thread may be placed round the neck.

To Cut Glass Tubing.—Make a cut at the desired place with a sharp triangular file, then place the file on the bench, rest the glass tube on the edge of the file with the cut uppermost, and press gently on each side of the cut. The glass tube will snap neatly in two pieces at the file-mark.

Platinum Wire.—This must be a piece about 10 cms. long. It is fixed in a piece of drawn-out glass tubing, by rotating the tube with the wire in position, in a Bunsen flame.

For borax beads a loop, A, is required. For flame coloura-

tions in which the solid is taken upon the wire, a loop, B, should be used (Fig. 15).

Stirring-rods.—These are made of glass rod, and they should be of such length, that a rod in use extends only one or two inches above the edge of the vessel used. Two of each size should be cut, namely, 7, 12, and 18 centimetres in length, and their ends rounded by rotation in the flame.

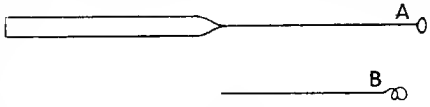

FIG. 15.

A rod should be used whenever a liquid has to be stirred, decanted, or filtered.

To Ignite a Precipitate and Burn a Filter.—When, in quantitative analysis, a ppt. has to be ignited and weighed, it must be transferred to a crucible, which is then mounted on a pipeclay triangle, as in Fig. 16. Ignition at a bright red-heat for 15 to 20 minutes is usually sufficient to drive off all traces of moisture, but it should be repeated until two consecutive weighings agree.


FIG. 16.—(*From Newth's "Manual of Chemical Analysis."*)

When the crucible has cooled slightly, it must be transferred to a desiccator by means of crucible tongs. When quite cool, it is removed from the desiccator and weighed.

Some of the ppt. will always adhere to the filter-paper, and this must be burnt by rolling the paper into a small roll with the ppt. wrapped inside. The platinum wire is then

wound round it (Fig. 17), and while held thus the filter-paper is burnt by placing it in a flame. It catches light, and is then withdrawn from the burner, and allowed to smoulder

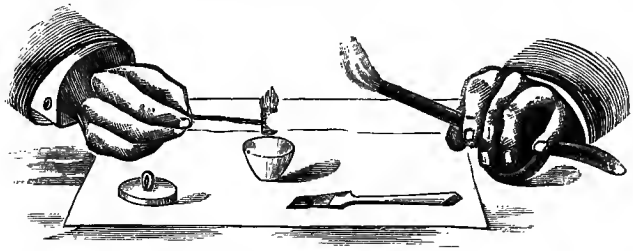

FIG. 17.—(*From Newth's "Manual of Chemical Analysis."*)

out. During this process, the burning filter must be held over the uncovered crucible, which contains the rest of the ppt. The crucible itself must rest on a sheet of glazed paper, so that any spilled portions may be collected without loss. When the filter has been completely burnt, the ash and the adhering ppt. are carefully tapped into the crucible. The weight of the ash, due to the filter-paper itself, must in all cases be deducted from the final weight.

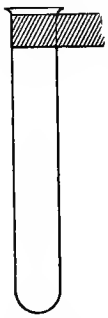

FIG. 18.

When the filter and ash need any special treatment beyond that mentioned here, full details will be given in the Quantitative Section.

Test-tubes are used for boiling or warming small quantities of liquid. For this purpose they may be held directly in the flame in a slanting position. Test-tube holders are often supplied to prevent burning the fingers, but a very serviceable and less clumsy holder can be made by wrapping a piece of post-card round the upper end of the tube, and holding the free ends together, as in Fig. 18.

Test-tubes are kept in a rack, in which they stand in an upright position. The best rack is one fitted with a wooden pillar opposite each hole, so that the tubes can be drained after washing.

All apparatus should be washed before finally placing in

the cupboard. It is *much easier and quicker* to wash out apparatus immediately after use than after standing for several days. The test-tube brush should be used for cleaning test-tubes and boiling-tubes. If water fails to remove stains, try in turn hydrochloric acid, nitric acid, or caustic soda. If the cold liquids are not sufficiently active, they should be heated.

To Cut Glass Tube of Wide Bore.—When tubing has a diameter greater than 1·5 cms. it is not easily broken, by the method explained on p. 12, especially if it be of hard glass. The following method must be adopted: Make a deep file-mark at the desired place, and in the direction which the break is required to take. Next make the end of a drawn-out piece of glass white hot, in the blowpipe flame, and place it quickly on the file-mark.

This procedure will start a crack round the tube, and if necessary the process may be repeated to complete the crack.

The Collection of Gases.—Gases may be collected in gas-jars, over water in a pneumatic trough (see Fig. 24), if they are not appreciably soluble in that liquid, *e.g.* oxygen and nitrogen. Those gases which are soluble in water to a marked extent must be collected by either upward or downward displacement of air. The upward displacement method (see Fig. 45) is suitable for gases lighter than air, such as ammonia; while downward displacement must be used (see Fig. 33) when the gas is heavier than air, *e.g.* chlorine and carbon dioxide.

When a jar is filled with the gas which is being collected, it must be covered with a glass plate which has been greased, in order to make the joint gas-tight.

The jar of gas may then be put aside in a cool place until it is required for use.

Kipp's Apparatus for Generating Gases.—This apparatus, shown in Fig. 19, should be used whenever possible for providing a steady stream of gas. The parts are as follows: The compartments B and C are made in one piece; the former contains the solid substance, and the latter acts as a reservoir for the acid used. The two compartments communicate by means of a narrow neck, and each one is fitted with a tubule and stopper.

The topmost part A consists of a large bulb, drawn out

below into a long, tapering tube, and when placed in position it fits perfectly into the ground neck of B, making a gas-tight joint.

The apparatus is charged for delivering a steady stream of CO_2 as follows: Small lumps of marble are introduced into B, while A is held loosely in position so as to prevent the marble falling into C. When B is half-filled, the upper part A is fixed in position and the hydrochloric acid is poured into it, while the tap *k* is kept open to allow the escape of air.

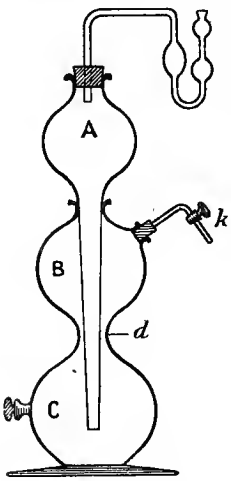

FIG. 19.

The acid runs into C, and the level is allowed to rise until it reaches the marble. The tap *k* is then closed, and a little more acid added. When *k* is closed and the acid attacks the marble, the pressure of CO_2 produced, forces the acid down until gas ceases to be evolved. In this manner B and part of C become filled with the gas, which forces the acid partly back into A.

The apparatus thus regulates itself, and whenever gas is drawn off from *k* the pressure falls, and acid is allowed to act on the marble once more.

Kipp's apparatus may be used for generating a steady stream of the following gases:—

Gas.	Solid Used.	Liquid Used.
Carbon dioxide.	Marble.	Hydrochloric acid (1 : 1).
Hydrogen.	Granulated zinc.	Sulphuric acid (1 part of strong acid to 4 parts water).
Hydrogen sulphide.	Iron sulphide.	Hydrochloric acid (1 : 1).
Hydrochloric acid (Hydrogen chloride.)	Lumps of ammonium chloride, or rock salt.	Concentrated sulphuric acid.
Chlorine.	Bleaching powder cubes.	Hydrochloric acid (1 : 1).

Sampling, Powdering, and Mixing.—In selecting a solid, a liquid, or a gas for analysis, great care must be exercised in obtaining a sample which represents the average properties of the whole.

For example, in dealing with a load of coal or mineral, lumps would be selected from different parts of the load to make sure that an average idea of the composition was obtained.

These specimen lumps would be ground and powdered to small size, thoroughly mixed, and laid out on a flat surface, making a square layer of several inches in depth. This would then be quartered and two opposite quarters selected, to be further powdered and mixed. This process of quartering and powdering would be repeated, till finally about a pound of the finely powdered substance was obtained. This example gives some idea of the care needed, and methods adopted for ensuring the collection of an average sample.

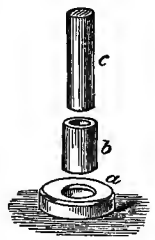

FIG. 20.—(*From Thorpe's "Quantitative Chemical Analysis."*)

In sampling peat or briquette fuels, the best plan would be to take borings from a few selected blocks.

In sampling water, a suitable and representative place must be selected for taking the sample, and the bottle used must be rinsed at least twice with this water before being finally filled.

In drawing off a sample of furnace gas, the gas must pass through the sampling vessel for a few minutes, or the latter must be filled two or three times over to ensure the driving out of all air.

Before attempting to dissolve any solid, it must be reduced to the finest powder possible, devoid of all grittiness. Lumps of mineral must be dealt with as follows: The lumps are first broken up into small pieces by wrapping in paper and repeatedly striking with a hammer. The smaller pieces so obtained are then transferred, a little at a time, to a steel mortar (Fig. 20). A is a solid block of steel, and into the hollow on the top, a steel cylinder B fits. The small lumps are placed in the cylinder, which is fixed in position on the steel block, and the solid steel pestle C is placed in the cylinder. This is struck repeatedly with a hammer, and by

this means the material is reduced to a coarse powder. The final powdering must be conducted in an agate mortar, using a pestle of the same material.

The powder ultimately used must be that which passes through fine muslin or cambric. The fabric is tied over a beaker of 10 cms. diameter, and the powder thrown on to it. By tapping with a glass rod, the finest particles will pass through, and that which remains on the muslin is returned to the agate mortar and re-ground.

Thorough mixing is in all cases a very necessary process. Students frequently fail to obtain desired results because they have neglected to mix the reagents thoroughly. When one liquid is added to another in a beaker or dish, mixing may be accomplished by use of a glass stirring-rod. If the reaction is carried out in a test-tube or boiling-tube, the mixing is accomplished by well shaking.

FIG. 21.—(*From Newth's "Manual of Chemical Analysis."*)

Similarly, when standard solutions are made up in measuring flasks, the contents must be well mixed by holding the stopper firmly and inverting the flask several times.

Weighing Precipitates in the Filter-paper.—When a ppt. is dried and weighed on the filter-paper in which it was filtered, it is necessary to dry and weigh the filter-paper before using it. This may be done by placing the folded paper in a tube fitted with a glass stopper (see Fig. 21). The tube, with the folded paper inside but without the stopper, is placed in the air-oven and heated for about forty-five minutes at 110° C. At the end of this time it is removed, the stopper inserted, and the whole allowed to cool in the desiccator before weighing. The weighed filter is then fitted into its funnel and the tube is left in the desiccator. When all the ppt. has been collected and dried completely in the air-oven, the filter is folded and slipped (with its precipitate inside it) into the glass tube, the stopper is re-inserted, and the whole allowed to cool in the desiccator before weighing.

Estimation of Moisture in a Substance.—The powdered specimen is best heated in an air-oven to the desired temperature, on a watch-glass. The glass is fitted with a cover, which takes the form of a second watch-glass. Both can be clipped together as shown in Fig. 22. The cover and clip are kept in the desiccator, and only fixed on to the glass which holds the substance, when it is cooling in the desiccator, and when it is weighed.

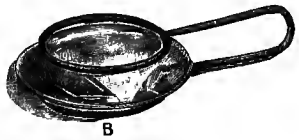

FIG. 22.—(*From Newth's "Manual of Chemical Analysis."*)

A Stoppered Weighing Bottle is to be used when a substance in the solid state is taken for quantitative analysis. The bottle can be filled with the finely powdered specimen, and for each determination a little is tipped out, and the weight taken is found by difference.

FIG. 23.—(*From Newth's "Manual of Chemical Analysis."*)

The Use of Symbols and Equations.—The chemical elements can be represented by symbols which are tabulated on p. 166. In most cases the symbol used is the first letter in the name of the element, while in other cases the symbol is made up of the first two letters of this name, or some suitable abbreviation of it. Occasionally, the symbol representing an element, is an abbreviated form of the Latin name, as for example:—

Copper = Cuprum,	has	the	symbol Cu
Silver = Argentum,	,,	,,	Ag
Gold = Aurum,	,,	,,	Au
Sodium = Natrium	,,	,,	Na
Potassium = Kalium	,,	,,	K
Mercury = Hydrargyrum	,,	,,	Hg
Antimony = Stibium	,,	,,	Sb
Iron = Ferrum	,,	,,	Fe

These symbols have a quantitative meaning. Each one, written alone, stands for the smallest particle of that element capable of entering into a chemical reaction, *i.e.* an atom.

Since to each atom a definite relative weight has been ascribed, the symbol represents also a weight of the element equal to the atomic weight.

For example: The symbol Cu represents one atom of metallic copper, and it also represents a total weight of sixty-three units, *i.e.* grams, &c.

In fixing the atomic weights given on p. 166, the atom of oxygen is selected for the standard, and is given the weight sixteen. In the metric system of measurement this will be sixteen grams.

Compounds are represented by formulæ, which give in a shorthand manner, the exact composition of each individual particle or molecule. Thus, $CuSO_4$ represents the fact that a molecule of the compound, copper sulphate, is composed of one atom of copper, one atom of sulphur, and four atoms of oxygen.

$2CuSO_4$ is written to represent two molecules of this substance, and $3CuSO_4$ represents three molecules. The number of molecules is always written in front of the formula and on the line. The number of atoms always follows the symbol, and is written below the line.

For example, $2O_3$ represents two molecules of ozone, each molecule being made up of three oxygen atoms.

Sometimes the formulæ are more complex, as in these examples: $Ca_3P_2O_8$, which represents one molecule of calcium phosphate, composed of three atoms of calcium, two atoms of phosphorus, and eight atoms of oxygen. This can also be written $Ca_3(PO_4)_2$, where the complex (PO_4) is treated as a chemical unit.

The formula, $2CuCO_3.Cu(OH)_2$, represents a molecule of basic copper carbonate, composed of two molecules of normal carbonate, intimately associated with one molecule of copper hydroxide. It is, so to speak, a trimolecular aggregate or molecule.

Chemical equations provide us with a shorthand method of stating the materials and their quantities, which enter into or result from a chemical reaction.

$2Hg + O_2 = 2HgO$, represents the fact that when mercury combines with oxygen gas to form mercury oxide, two atoms of the metal combine with two atoms of oxygen (one molecule), to form two molecules of mercury oxide.

Further, since the atomic weights are: mercury = 200, oxygen = 16, the equation states that 400 gms. of mercury will require 32 gms. of oxygen, and the weight of oxide formed will be 432 gms.

Similarly the reaction between calcium carbonate and hydrochloric acid is represented thus:—

$$\begin{array}{ccccccccc} CaCO_3 & + & 2HCl & = & CaCl_2 & + & CO_2 & + & H_2O. \\ \underbrace{40+12+48} & + & \underbrace{2(1+35{\cdot}5)} & = & \underbrace{40+71} & + & 44 & + & 18. \\ 100 & + & 73 & = & 111 & + & 44 & + & 18. \end{array}$$

In which we state that, to completely decompose 100 gms. of carbonate, we shall need 73 gms. of hydrochloric acid. The resulting products will be 111 gms. calcium chloride, 44 gms. carbon dioxide, and 18 gms. of water.

The correct writing of formulæ and chemical equations is largely the result of practice, and the student is urged to write down equations for every reaction which he meets. While most of the work in this book can be followed without any knowledge of equations or formulæ, it is impossible to follow, with intelligence, the analytical sections of the work without such knowledge.

The terms "molecular proportion" and "atomic proportion" are sometimes used in place of "molecular weight" and "atomic weight."

CHAPTER I

PHYSICAL CHANGE AND CHEMICAL CHANGE— PHYSICAL MIXTURES AND CHEMICAL COMPOUNDS

When a substance undergoes a physical change its molecular composition remains unaltered.

Any change which alters its molecular composition is described as chemical.

Exp. 1.—Hold a piece of glass tube in the Bunsen flame, and notice that after becoming red hot it melts. Remove it from the flame and observe that, when cool, it has all the appearance and properties of the original glass.

Exp. 2.—Magnetise a piece of knitting-needle by stroking it a few times along its length, in one direction, with one end of a bar magnet.

The needle-piece now exhibits the properties of a magnet, *i.e.* it attracts iron filings and sets in a north and south direction when suspended horizontally. It remains, however, a piece of steel, and is identical in appearance and properties with the original unmagnetised piece.

***Exp.** 3.—Place, in a small evaporating dish, as much cane sugar as would cover a halfpenny. Add three or four times the bulk of warm water and stir well.

The cane sugar disappears completely, and in its place is found a clear solution.

Evaporate this gently over a very small flame until the water has just evaporated.

The cane sugar remains behind, equal in quantity to, and identical with, that originally used.

These three experiments provide us with examples of "physical change."

***Exp.** 4.—Heat the sugar remaining in the dish, strongly. Notice that it soon melts and then chars, evolving at the same time smoke and fumes.

The sugar has undergone chemical change, and in its place is left a small quantity of black carbon.

Exp. 5.—Heat a small piece of magnesium ribbon in the flame, as you did glass in Exp. 1. The metal burns with a brilliant light, and left behind in its place is a white ash totally different to the metal used. The magnesium has undergone a "chemical change."

Having ascertained by this work the distinction between physical and chemical change, carry out the following experiments to learn the difference between a physical mixture and a chemical compound :—

***Exp. 6.**—(*a*) Weigh approximately 20 gms. of iron filings on a filter-paper.

(*b*) Weigh approximately 12 gms. of powdered sulphur on a filter-paper.

Transfer both to an evaporating dish and mix thoroughly with a glass rod.

When the mixing is complete, divide the powder into two nearly equal portions, A and B.

Taking portion A, note the dark grey powder, looking different from either of the original substances used. That it is really not different, but is only a mixture, may be shown as follows :—

(1) Examine a little with a lens. Particles of iron and sulphur are discernible.

(2) Draw a magnetised knife-blade through another small quantity. Particles of iron are drawn out, and by repeated attraction the filings are completely removed.

(3) Place a small quantity in a beaker, and by pouring on to it water, and stirring well, wash the sulphur away from the heavier filings by pouring off the water. This is an example of a "physical mixture," and the percentage of each ingredient could be estimated by either the magnetised knife-blade, or by washing away the sulphur. However, carry out this estimation by dissolving the iron filings with hydrochloric acid (hydrogen chloride), in which solvent sulphur is quite insoluble.

***Exp. 7.**—Weigh accurately about 10 gms. of portion A of the mixture, on a small weighed filter-paper (or counterbalance the filter-paper by another similar one).

Transfer this completely to a 150 c.cm. beaker, and pour on

gradually about 80 c.cms. of strong hydrochloric acid (1 : 1). The iron filings dissolve with effervescence, and when all the acid has been added, stir the liquid for fifteen minutes to ensure complete solution of the iron filings.

Filter the solution through the paper which was weighed (or counterbalanced), and make sure that all the sulphur is ultimately placed in the filter-paper.

When all the iron solution has run through, wash the filter with hot water, and finally transfer the drained filter containing the sulphur into the hot-water oven to dry. When quite dry, weigh, and from the weight of sulphur found calculate the percentage of iron and sulphur in the mixture. Compare the results with the weights actually taken at first, when the mixture was made. In this experiment a physical mixture has been made, and two or three ways of separating the ingredients shown, and lastly, the percentage of each ingredient has been estimated by dissolving one from the other.

***Exp. 8.**—(To be performed while the filter of Exp. 7 is drying). Portion B, which was set aside, is now placed in a test-tube.

Heat the tube containing the mixture of iron and sulphur, gently, in the flame.

The contents soon begin to glow, give off sulphurous fumes, and show every sign of a vigorous interaction.

When the action has moderated, set the tube aside to cool. After cooling, knock the contents of the tube into an evaporating dish, breaking the tube if necessary.

If the glass is broken, pick out all pieces of glass as far as possible, then powder the grey mass.

Observe the dark grey powder, somewhat like the mixture it was, before subjecting it to heat.

The following experiments, however, show that it is totally different :—

(1) In a small portion, no separate particles of iron and sulphur are discernible with a lens.

(2) The magnetised knife-blade no longer draws out any filings, showing that the new substance is entirely non-magnetic.

(3) It is no longer possible to separate the sulphur from the iron by washing it away (levigation), because all the particles have the same density.

***Exp.** 9.—Place a small quantity in a test-tube and pour on to it some dilute hydrochloric acid.

Observe :—

(1) The mass completely dissolves, except possibly a small trace of sulphur (white).

(2) The gas evolved with effervescence has a very unpleasant odour, and makes a lead acetate paper black when brought into contact with it.

It is no longer possible to estimate the percentage of iron and sulphur present, by the "simple solution" method used in Exp. 7, nor by any other purely mechanical or physical method.

Only chemical methods may be used, involving the "decomposition" of the substance.

We are here dealing with a "chemical compound" formed by the "union" of two elements under the influence of heat. The equation representing the reaction is—

$$\underset{\text{Iron.}}{Fe} + \underset{\text{Sulphur.}}{S.} = \underset{\text{Iron Sulphide.}}{FeS.}$$

CHAPTER II

COMPOSITION OF AIR AND WATER—OXIDATION AND REDUCTION

***Exp.** 1.—(*a*) Heat in a crucible a small piece of metallic lead. Note that the lead melts, and if the heating is continued for five minutes the bright metallic surface becomes coated with a reddish-yellow dross.

(*b*) Heat a piece of copper wire in the flame. It becomes red hot, and on removal from the flame and cooling it will be seen to have turned black. Scrape off this black coating with a knife and note the bright red copper beneath.

Nearly all metals become covered with a dross (or oxide as it is termed) when strongly heated in air, and a few metals will even burn (magnesium).

In every case the weight of the dross is greater than the weight of the original metal.

***Exp.** 2.—Weigh in a crucible, accurately, about 0·2 gm. magnesium ribbon. Put on the lid, and heat the crucible on a pipeclay triangle. When the crucible is just red hot, remove the lid for a second or two with crucible tongs and replace.

Note how the metal is burning and glowing, particularly when it is well exposed to the air by removal of the lid.

After heating in this way for fifteen minutes, and removing the lid at intervals, take away the burner and let the crucible cool.

When quite cold, weigh the crucible with lid and contents, and estimate the percentage gain in weight.

Enter the result in your note-book thus :—

Weight of crucible + lid + magnesium =
" " " + dross of magnesium =

Gain in weight of magnesium =

Many of the non-metallic elements burn in air similarly, forming a dross (or oxide). Such are carbon (coal, coke), sulphur, and phosphorus. In these cases, however, the dross is volatile, and escapes as a smoke; but if the dross is collected, in each case there is a gain in weight shown by burning the element in air. It can be assumed, therefore, that when elements burn or are heated in air, they combine with part of the air and form drosses or oxides.

*Exp. 3. To find out what Part of the Air is used up in Burning or Calcining.

Take a glass tube, about 50 cms. long, and having an internal diameter of between 1 and 2 cms. The tube should be sealed at one end only, and form therefore what might be regarded as a very long test-tube.

The open end should be fitted with a rubber cork, so that when needed, the open end may be closed.

Place in the tube a small piece of asbestos fibre, and then push it down to the lower end. This forms a bed, on which a piece of phosphorus, the size of a pea, can be dropped, and the rubber cork quickly inserted.

To burn the phosphorus in the air enclosed in the tube, dip the end holding the phosphorus in a beaker of hot water. The phosphorus soon melts and catches fire. When it takes fire, remove the tube from the hot water and allow the phosphorus to burn out. When this has taken place, and the tube is cool (ten to fifteen minutes), remove the rubber cork, while the corked end is under water standing in a gas-jar. Finally, hold the tube so that the water which enters it, is at the same level as that in the jar outside, and measure the length of tube containing residual air.

Raise the tube carefully, and insert the cork while the open end is still under water. Put the corked tube on one side for further investigation.

What volume of air has been used up in burning the phosphorus? If worked through correctly, almost exactly one-fifth of it will have disappeared.

Now find out what kind of air fills the remaining four-fifths of the tube. It is colourless, like ordinary air.

Hold the tube vertically so that the water goes to the

closed end, and, removing the cork, quickly insert a lighted taper. It is immediately extinguished.

Conclusions.—Air is composed of two parts. One part supports combustion, and combines with burnt or heated substances to form drosses or oxides. This forms approximately one-fifth by volume of the air.

The other part, making up four-fifths, is inactive as far as combustion is concerned, for it will not keep a burning taper alight.

This inactive part has been named *nitrogen*, while the active part which supports combustion and combines with heated metals to form "dross" is called *oxygen*.

*Exp. 4. To Burn Copper in Air and examine the Residual Gas left behind.

Arrange the apparatus as in the figure. It is made up of the following parts:—

A is a Winchester-quart full of air. When water is poured into the funnel of the long tube which reaches the bottom, air is driven out into B, which contains metallic copper heated to redness. It is made of combustion tubing 20 cms. long, 1 to 2 cms. diameter. The ends of B are closed by single-bored rubber corks, one of which is pierced by the short tube from A, while the other is pierced by a delivery tube which dips under the surface of water in the pneumatic trough C.

A gas-jar filled with water is placed on a beehive-shelf to collect escaping gas (see p. 15).

The funnel at K should be joined on to the long water-tube by six inches of rubber tube, on which a clip may be used to regulate the flow of water. The funnel itself can be supported by a retort-stand ring. A large vessel of water may advantageously replace the funnel. This vessel will stand on a higher level than K, and be connected at this point by a syphon tube.

Heat the tube containing the copper turnings to a dull red heat, being careful not to burn the rubber corks on each side. Each cork may be protected by a small square of asbestos sheet, *m m* (see Fig. 24). When the copper is red hot, allow the water to run down into the "Winchester" at

such a rate that bubbles of gas issue through the water in C in fairly rapid succession, and collect two jars of this gas. Note that the copper has become tarnished and black, just as it did when heated in air. Now disconnect at *b* and

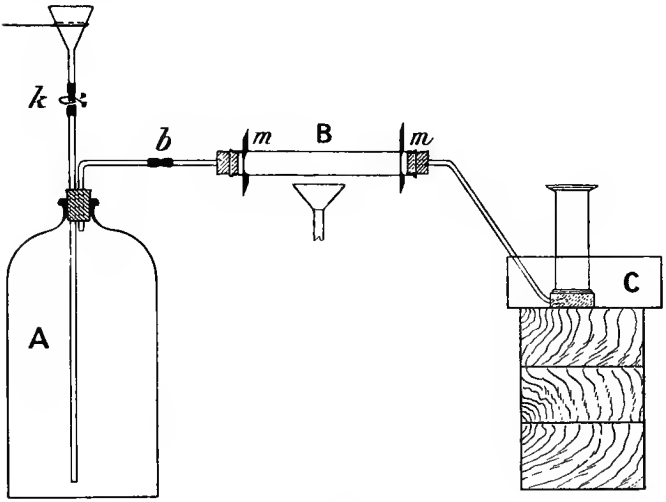

FIG. 24.

remove the burner. Test the two jars of gas collected as follows :—

(*a*) Thrust into one a burning taper, and note that it is extinguished.

(*b*) Thrust into the second jar a piece of burning magnesium ribbon (held in crucible tongs). Note that this likewise is extinguished.

This experiment supports the conclusions already reached in Exp. 3, that air consists of two parts, viz. active air or oxygen, and inactive air or nitrogen.

The chemical equations representing the oxidation of the elements so far used are :—

(1) $2Cu + O_2 = 2CuO.$

Copper. (2 atoms) Oxygen. (1 molecule) Copper oxide. (2 molecules)

(2) $2Mg + O_2 = 2MgO$.
Magnesium. Oxygen. Magnesium oxide.

(3) $4P + 5O_2 = 2P_2O_5$.
Phosphorus. Oxygen. Phosphorus pentoxide.

Air is a *mixture* of oxygen and nitrogen, and not a compound. One important proof of this is, that the two ingredients can be separated by physical means, namely, *fractional distillation* (see p. 85).

In the neighbourhood of $-190°$ C., air is a liquid, and if the temperature is allowed to rise gradually, the nitrogen, which is more volatile, boils off at $-193°$ C. The liquid oxygen left behind in the distilling vessel does not boil off until the temperature has risen to $-182{\cdot}5°$ C.

Exp. 5. To Prove that the Rusting of Iron is only a Special Case of Slow Oxidation.

Take the glass tube used in Exp. 3 (p. 27), tip into it as much of damped iron filings as will well cover a penny piece. Insert the rubber cork securely, and shake the tube so that the filings become distributed over the inner surface. Leave this for twenty-four hours, and then examine. Notice that the filings have acquired the reddish colour of rust. Open the tube under water in a large beaker, as was done in Exp. 3, and observe that some of the air has been used up, as shown by the rise of water in the tube. Adjust the water levels and register the volume of air used up. Re-insert the cork while the open end is still under water, and again put the tube aside. Repeat these observations after another twenty-four hours, and repeat at daily intervals until no further rise in level is registered. The final measurement will show that the filings in rusting have used up one-fifth of the original air volume. The oxygen has in this case oxidised the iron slowly to rust. This reaction is represented by the equation—

$4Fe + 3O_2 = 2Fe_2O_3$.
Iron and oxygen. Red iron oxide.

Invert the tube, and thrust into the residual gas a lighted taper or burning magnesium. Either of these is extinguished.

Exp. 6. First Step to Elucidate the Composition of Water.

Fit up the apparatus shown in Fig. 25. It consists of a half-litre flask A, fitted with a single-bored cork, through which a right-angled glass tube leads to a short piece of combustion tubing, as used in Exp. 4, p. 28. From this, a gas-delivery tube leads under the water in a pneumatic trough. The combustion tube is packed loosely with iron borings. To start with, the flask should be disconnected at *b*, where it joins on to B by rubber tube.

Heat the water in the flask (half-full) to boiling,[1] and

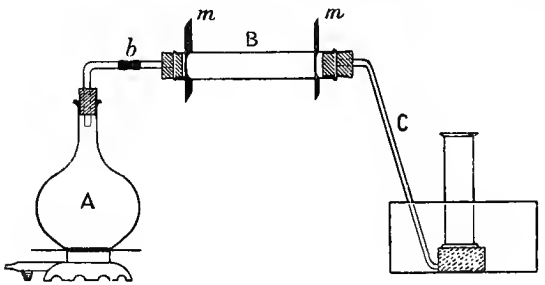

FIG. 25.

while the water is being heated, place a small flame under B. This flame can be increased gradually until the tube is at a dull red heat.

When a steady jet of steam is issuing from the flask, connect at *b*. A current of steam is thus driven over the red-hot iron, and bubbles of gas will escape through the delivery-tube in the pneumatic trough. Allow the first few bubbles to escape, and then invert a gas-jar filled with water, over the beehive-shelf and collect the gas which is issuing. Place this jar, when full of gas, on one side, and bring over the delivery-tube a second jar, half-full of water. When the water has been displaced, this jar will contain a mixture of approximately one volume of air and one volume of the gas

[1] A small piece of porous pot in the flask will promote *regular* boiling.

which is being collected. Remove this jar and place it on the bench with a greased glass plate covering it. Now light a taper, remove the glass plate, and apply a light to the open end of the jar. A loud report denotes that the gas being collected forms an explosive mixture with air.

Now take the jar which was previously filled with the gas, and while it is inverted remove the glass plate and introduce a lighted taper. There is no explosion, but the gas burns quietly with a blue flame, at the mouth of the jar. On pushing the taper right in, it is extinguished.

The conclusion from these experiments is, that the gas is inflammable, but does not support combustion—quite an opposite state of affairs to that presented by oxygen.

The gas is called hydrogen, and its extreme lightness may be proved by filling a third jar and allowing it to stand for one minute, without any cover, mouth upwards.

Apply a light at the end of this time. There is no evidence of hydrogen gas, it having escaped upwards into the air.

Now disconnect the steam flask at *b* and withdraw the sources of heat.

Question: Where did the hydrogen come from?

It may be assumed, for the present, that it came from the steam; that the steam by its passage over heated iron has been decomposed, and that one of its constituents has been isolated—namely, hydrogen.

Further, if the iron borings were previously bright, it will now be observed that they are tarnished. This tarnish *may be* similar to that produced when bright metals are heated in air, and if so, the iron borings have become oxidised.

*Exp. 7. To Pass Hydrogen over Heated Copper Oxide and Collect the Resulting Product.

For this experiment fit up the apparatus shown in Fig. 26. A is a generating flask, in which hydrogen is to be prepared by the action of sulphuric acid on zinc. B is a washing-bottle containing a little concentrated sulphuric acid, which serves to dry the hydrogen passing through. C is a combustion tube 20 cms. long, filled with granulated copper oxide, and fitted at each end with single-bored rubber corks. The bent

tube D enters a small flask, which is kept cool by immersion in cold water.

Note.—Hydrogen has already been prepared by the action of steam on heated iron, and it is known that metallic sodium decomposes cold water, liberating hydrogen.

This reaction between water and common metals is quite general, but it is usually necessary to add some acid or alkali to the water in order to facilitate the decomposition.

Cover the bottom of the flask A with granulated zinc, and insert the cork holding a thistle-funnel and right-angled tube. Join the right-angled tube to the washing-bottle B, and complete the connections as in Fig. 26.

Pour sufficient dilute sulphuric acid down the thistle-funnel

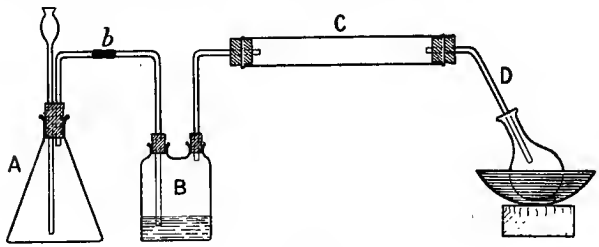

FIG. 26.

to cover the zinc. (A Kipp's apparatus (see p. 16) may be used instead of the flask A.)

The acid soon reacts with the zinc, and a steady stream of gas is evolved. Allow the gas to pass through the apparatus for two minutes, and then collect a test-tube of it and bring it near a flame to ascertain whether all air has been expelled. If this is so, the hydrogen will burn quietly. The test-tube may be filled by bringing it mouth downwards over the end of the tube D.

When it is certain that all air has been expelled, place a small flat flame under C and commence heating the copper oxide. Gradually increase the heat until the tube becomes red hot, and meanwhile place a small receiving flask to catch any liquid product passing down D.

A steady current of hydrogen must be maintained by adding fresh acid occasionally.

When between 10 and 20 c.cms. of colourless liquid has collected in the receiver, stop the process by removing the flame from the copper oxide tube and let it cool down while a slow stream of hydrogen gas passes through.

After a few minutes, stop the current of hydrogen by disconnecting at *b*, the generating flask.

Now examine the liquid product in the receiver.

It is colourless. Taste a drop and compare the taste with that of distilled water.

Add a few drops to some white anhydrous copper sulphate in an evaporating dish, and compare the result with that obtained when a few drops of distilled water are added to another portion of anhydrous copper sulphate.

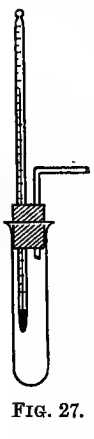

FIG. 27.

Lastly, determine the boiling point of the liquid and compare it with that of pure water (100° C.). The boiling point determination is carried out in the apparatus shown in Fig. 27. It consists of a test-tube fitted with a double-bored cork. Through one hole a thermometer is inserted so that the bulb reaches to within 2 inches of the liquid surface. The liquid in the tube should be from 1 to $1\frac{1}{2}$ inches deep, and contain a piece of porous pot. Place it on a sand-bath and heat with a small flame. The liquid ultimately boils, and the steam escapes through the open hole in the cork. Mark the temperature finally maintained by the thermometer while the liquid in the tube is gently boiling.

All these observations prove, that the liquid obtained by passing hydrogen gas over heated copper oxide, is pure water.

Note that the black oxide has been partly changed to red metallic copper, and it must therefore be concluded that the hydrogen has combined with the oxygen, and the resulting compound formed is water.

Equation—

$$\underset{\substack{\text{Hydrogen}\\(2\text{ molecules})}}{2H_2} + \underset{\substack{\text{Oxygen}\\(1\text{ molecule})}}{O_2} = \underset{\substack{\text{Water}\\(2\text{ molcules}).}}{2H_2O}.$$

The reactions carried out in these last experiments are represented by the following equations :—

1. Passing steam over red-hot iron :

$$3Fe + 4H_2O = Fe_3O_4 + 4H_2.$$

2. Preparation of hydrogen from zinc and dilute sulphuric acid :

$$Zn + H_2SO_4 = ZnSO_4 + H_2.$$

3. Passing hydrogen over copper oxide :

$$CuO + H_2 = Cu + H_2O.$$

*Exp. 8. To Prepare Oxygen Gas by heating Oxide of Mercury.

Certain oxides or "rusts" part with their oxygen on being

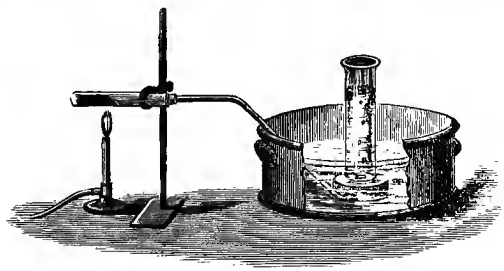

FIG. 28.—(*From Jago's "Elementary Inorganic Chemistry."*)

heated to a sufficiently high temperature. Oxide of mercury is one of these.

Place about 20 gms. of red mercury oxide in a hard glass tube, fitted with a single-bored cork and delivery-tube. Clamp it as shown in Fig. 28, and let the exit end of the delivery-tube dip under a bee-hive shelf, which is under water in a pneumatic trough.

Heat the tube, at first with a smoky flame and then with the blue flame obtained by turning the air-valve at the base of the burner. Bubbles of gas soon commence to pass through the water. After the first few have escaped, place an inverted gas-jar filled with water, on the bee-hive shelf, so that the gas passes up and is collected in the jar.

In this way collect three jars of oxygen, and place them on the bench, each covered with a greased glass plate.

Note the globules of mercury which have collected on the side of the tube. The red oxide has been decomposed by heat, into oxygen and mercury.

Equation—

$$\underset{\text{Oxide of mercury.}}{2HgO} = \underset{\text{Mercury.}}{2Hg} + \underset{\text{Oxygen.}}{O_2}.$$

Carry out the following experiments with the three jars of oxygen :—

(*a*) Light a wood splinter. Blow it out, and, while it is glowing only, plunge it into the jar of oxygen. Does oxygen support combustion readily?

(*b*) Heat a small piece of carbon to redness in a deflagrating spoon, and when it is glowing, plunge it into the second jar. Note the brilliance with which the combustion is continued. When the jar has cooled somewhat, pour in about 20 c.cms. of distilled water; close the jar with a glass plate and shake well. Pour half of this water into a tube containing a little clear lime-water, and note the turbid appearance. Pour the other half into a tube containing a little blue litmus solution, and note the deep red colour given.

(*c*) Heat a little sulphur in a deflagrating spoon until it burns, and while it is burning, plunge it into the third jar of gas. Note the increased brilliancy of combustion, and, when the jar is cool, pour in a little blue litmus solution. Note the pink colour given to the litmus, and also observe the choking acid odour of the gas produced.

In the last two cases the colourless and odourless oxygen has oxidised carbon and sulphur to acid substances soluble in water, their acid nature being shown by their ability to turn litmus solution red.

Oxidation takes place when an element combines with oxygen, and the resulting compound is termed an oxide.

Reduction is the opposite of oxidation, and implies the removal of oxygen from a compound.

In Exp. 7 (p. 32), copper oxide was reduced to metallic copper by the action of hydrogen.

Practically all oxides when heated in a current of hydrogen gas, become reduced; the hydrogen combining with the oxygen in the oxide to form water.

***Exp. 9. To Prove that Carbon is a Reducing Agent.**

Take a charcoal block, and in it scoop a shallow basin having a diameter of about 1 cm.

Place in this scoop a small quantity of red lead (oxide of lead). Now, by means of a mouth blowpipe, direct a small flame on to the powder, and make it red hot. The red powder darkens in colour, and, after a moment or two, a bead of soft metallic lead will be found at the bottom. The oxide has been reduced to lead by the action of heated charcoal. (See Fig. 11.)

Repeat this experiment, with a fresh scoop each time, taking in turn iron rust (oxide of iron), tin oxide (tin stone), and copper oxide, and using the reducing flame.

Iron oxide will give a black magnetic powder (iron). Tin oxide will give a bright metallic bead of tin. Copper oxide will give red particles of metallic copper.

Carbon is the reducing agent used so largely for reducing metallic ores. In this case the oxides are roasted in suitable furnaces with coke or anthracite coal.

CHAPTER III

OXIDES AND HYDROXIDES—BASES, ACIDS, AND SALTS

(1) WHEN an oxide combines with water it forms a hydroxide.

(2) Oxides are of two kinds, basic and acid.

(3) When a basic oxide combines with an acid oxide the product is a salt.

***Exp. 1. To Prepare Calcium Hydroxide (Slaked Lime).**

Weigh accurately, in a small evaporating dish, about ten gms. of lime (calcium oxide).

Add a little distilled water to it, and break it up gently with a glass rod. Notice the large amount of heat evolved during this process (slaking), a sign that chemical action is proceeding.

When, by adding more water and stirring, a thin paste is obtained, evaporate the excess of water on a water-bath, and when the powder appears quite dry, place the dish in a desiccator to cool, and after cooling take the weight. An increase in weight will show the amount of water which has combined with the lime, and the weighings should be entered thus:—

A. Weight of empty dish =
B. Weight of dish + lime =
Weight of lime (CaO) =

After adding water and drying—

C. Weight of dish + calcium hydroxide =
Gain in weight due to water (C − B) =

Do these results agree with the equation?

$CaO + H_2O = Ca(OH)_2$.

56 gms. + 18 gms. = 74 gms., or gain of 32 per cent.

Exp. 2.—Shake up some of this calcium hydroxide with distilled water. Some of the solid dissolves, and on placing a piece of red litmus paper in the solution, the colour will change to blue.

Substances which, when dissolved in water, give solutions which turn red litmus blue, are termed alkalis and are said to be alkaline.

Alkaline oxides are always basic.

***Ex.** 3.—Place in three test-tubes a few drops of :—

(*a*) Potassium hydroxide solution, in tube 1.
(*b*) Sodium hydroxide ,, ,, 2.
(*c*) Ammonium hydroxide ,, ,, 3.

Put into each of these tubes a few drops of neutral litmus solution (or a piece of red litmus paper), and note the resultant colour change.

Evidently these three hydroxides are alkaline and basic. The corresponding oxides of sodium and potassium are the strongest bases known. When they combine with water the hydroxides are formed.

Equations—

$$\underset{\text{Sodium oxide.}}{Na_2O} + H_2O = \underset{\text{Sodium hydroxide.}}{2NaOH}.$$

$$\underset{\text{Potassium oxide.}}{K_2O} + H_2O = \underset{\text{Potassium hydroxide.}}{2KOH}.$$

***Exp.** 4.—Pour into three test-tubes a small quantity of solution containing :—

(*a*) Sulphuric acid, in tube 1.
(*b*) Hydrochloric acid, ,, 2.
(*c*) Nitric acid, ,, 3.

In each of these place a piece of blue litmus paper, and observe the red colour which appears.

Note.—Acids redden litmus paper.

Now drop into each one a piece of granulated zinc, and warm gently. In each case the zinc is dissolved with effervescence.

Next take three clean test-tubes and pour in a fresh quantity of the three acids. Now drop into each a small piece of washing soda (sodium carbonate).

Observe that the solid dissolves with effervescence. The properties of acids in solution are therefore:—

(1) They redden litmus.

(2) They dissolve zinc (and most metals) with effervescence.

(3) They dissolve sodium carbonate with effervescence.

Note.—In Exp. 8, p. 35, it was found that the oxides of carbon and sulphur dissolved in water, giving acid solutions. This general rule is valuable, namely, the oxides of the metals are basic, while the oxides of the non-metals are acidic.

*Exp. 5. To Make a Salt by combining Base with Acid—Neutralisation.

Measure out by means of a pipette, 25 c.cms. of 20 per cent. sodium hydroxide solution. Run it into an evaporating dish, add a few drops of litmus solution, and then run in carefully some 20 per cent. hydrochloric acid solution, stirring with a glass rod all the time.

As soon as the blue colour changes to a *faint pink* the acid has exactly neutralised the base, and the salt in solution will be sodium chloride (common salt). To obtain crystals of the salt, place the dish upon a water-bath and evaporate until a crust of crystals collects on the top of the hot liquid. Then remove the dish, and, on cooling, a considerable quantity of crystalline salt will be obtained. Drain off the excess of cold water and then place in the steam oven to dry.

Equation—

$$\underset{\text{Sodium hydroxide.}}{NaOH} + \underset{\text{Hydrochloric acid.}}{HCl} = \underset{\text{Sodium chloride.}}{NaCl} + H_2O.$$

Exp. 6. To Prepare Copper Sulphate.

Measure into a small beaker about 100 c.cms. of 20 per cent. sulphuric acid solution. Place it on a wire gauze and warm. To the warm acid add about 16 gms. of black copper oxide. The oxide will dissolve in the acid, and ultimately, a drop removed on a glass rod from the beaker will not redden a piece of blue litmus. The solution is then neutral, blue in colour, and contains copper sulphate.

If the copper oxide all dissolves and the solution is still acid, add a little more copper oxide.

In any case there must be a small amount of undissolved oxide, from which the neutral blue liquid must be filtered into an evaporating dish. When the filtration is completed, place the dish upon a water-bath and evaporate to crystallisation as was done in the preparation of common salt.

To obtain large crystals of copper sulphate, the hot liquid should be placed in the cupboard, and allowed to cool down slowly without any shaking.

Note.—Many of the minerals of commercial importance are metallic oxides, and these are tabulated with the other important minerals on page 170.

Water, the oxide of hydrogen, is certainly to be regarded as the most important of oxides. Its wide use as a solvent, and its value in the form of steam, as a source of power, show it to be of the greatest importance. It is further significant, that most chemical changes take place, only with difficulty, if every trace of moisture be removed from the reacting substances.

The characteristic properties of bases and acids are only exhibited in the presence of water, and frequently their corrosive action upon metals depends on the water present.

The table on p. 178 shows to what extent various metals and alloys are attacked by water itself, and water which contains various acids, alkalis, or salts in solution.

In some cases it will be noticed that the pure acid is without action upon certain metals.

The following experiment should be carried out in connection with this subject.

***Exp. 7. To Investigate the Action of Acids and Alkalis in Aqueous Solution upon the Metals, Zinc, Iron, Lead, Tin, Copper, and Aluminium.**

The trials should be conducted in the apparatus shown in Fig. 29; both acids and alkalis should be used in strong and weak solutions. The action should be tried with and without heating, and it will be well to carry out the work in the order and manner indicated in the table drawn up below.

The apparatus consists of a flask having about 100 c.cms. capacity, fitted with a single-bored cork, through which passes the bent delivery-tube. Any gases evolved can be collected in an inverted test-tube over water which stands in a small evaporating dish.

When it is necessary to heat the flask on a tripod, the

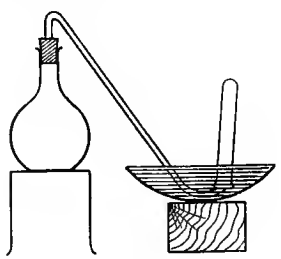

FIG. 29.

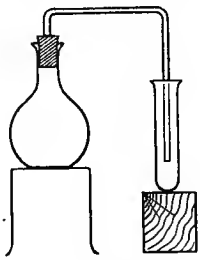

FIG. 30.

evaporating dish can be correspondingly raised on a box or wood-block.

Draw the table (p. 43) in your note-book, and in the spaces provided enter the following observations:—

1. Whether the metal dissolves or not.
2. Whether any gas is evolved and collected.
3. Whether the gas is—
   - (*a*) Inflammable.
   - (*b*) Supporter of combustion.
   - (*c*) Acid to litmus.
   - (*d*) Possessed of any smell.

N.B.—(1) In each case use as small a flask as possible.

(2) When there is a vigorous action in the cold it is *not* necessary to heat at all.

(3) Only 1 to 2 gms. of metal should be used in each experiment, and about 20 to 30 c.cms. of acid.

(4) If heated at all, the solution should not be raised in temperature beyond gentle boiling.

(5) The weak solutions should contain about 5 per cent. of the reagent.

The strong solutions should contain about 30 per cent. of the reagent.

	Zinc.		Iron.		Lead.		Tin.		Copper.		Aluminium.	
	Cold.	Hot.	Cold.	Hot.	Cold.	Hot.	Cold.	Hot.	Cold.	Hot.	Cold.	Hot.
* Sulphuric Acid. Dilute												
* Sulphuric Acid. Strong .												
Hydrochloric Acid. Dilute .												
Hydrochloric Acid. Strong .												
Nitric Acid. Dilute												
Nitric Acid. Strong . .												
* Sodium Hydroxide. Dilute .												
* Sodium Hydroxide. Strong .												

Note.—In the action of strong hot sulphuric acid on metals, any gas evolved should be collected by downward displacement in the apparatus of Fig. 30.

***Exp. 8. To Prepare Copper Oxide by Precipitation.**

Weigh accurately about 30 gms. of powdered copper sulphate (blue vitriol). Dissolve this in about 100 c.cms. of distilled water in a beaker, and, to facilitate solution, heat the vessel on a tripod and stir with a glass rod. When the solid has completely dissolved, let the liquid boil gently, and avoid spirting of the contents by covering the beaker with a small clock-glass. When the liquid is boiling, remove the burner and take off the clock-glass, and then pour in slowly some sodium hydroxide solution whilst stirring well. The quantity of soda required will be about 50 to 60 c.cms. of a 20 per cent. solution. At first a green-blue ppt. will be produced, which rapidly changes to black, and when all the $CuSO_4$ has been precipitated, the liquid must give a deep blue colour to a piece of red litmus. The black ppt. is copper oxide, and the green-blue ppt. observed at first is copper hydroxide. Near the boiling-point of water this latter substance loses the elements of water according to the equation

$$Cu(OH)_2 = CuO + H_2O.$$

When the liquid has been stirred, filter it through a weighed (or counterpoised) filter-paper, and when all the ppt. has been transferred to the filter, wash it twice with a stream of hot water from a wash-bottle. Then place the funnel and ppt. in a tin cone (see p. 9) to dry, and when quite dry, weigh it.

Calculate the percentage of black CuO obtained from the weight of copper sulphate taken. It should be, if nothing has been lost, 32 per cent.

The Peroxides of the Metals are bodies containing a higher percentage of oxygen than ordinary oxides.

The commonest examples are: Red lead, barium peroxide, manganese dioxide, and lead peroxide.

When strongly heated, these bodies evolve oxygen and become reduced to a lower state of oxidation.

Barium peroxide was for long used in the commercial preparation of oxygen gas, and at the present time it is used for making the liquid oxidiser and bleacher known as peroxide of hydrogen.

The peroxides, when heated with strong hydrochloric acid, oxidise the acid, liberating chlorine, which can be recognised by its characteristic smell and bleaching action.

This property of peroxides will be again referred to in Chapter VI., which deals with chlorine and its compounds (see p. 60).

CHAPTER IV

DETERMINATION OF CHEMICAL EQUIVALENTS

*Exp. 1. To Find the Equivalent of a Metal by Treating it with an Acid.

(The equivalent of an element is that weight which combines with or replaces 1 gm. of hydrogen.)

Equivalent of Zinc.—Weigh accurately from 0·6 to 0·8 gm. of zinc and place it in the flask of Fig. 31. Cover it with water and fill the small tube with strong hydrochloric acid. When the acid is allowed to come into contact with the zinc, hydrogen will be evolved, and may be collected in the apparatus here shown.

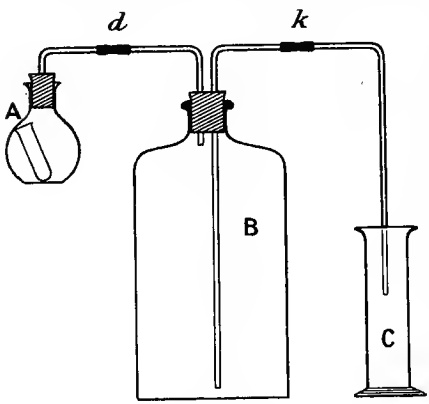

FIG. 31.

The vessel B, a Winchester-quart, acts as a syphon, so that when hydrogen is evolved in A the pressure acting in B will drive water over into the gas jar C. The volume of water collected here will be a measure of the volume of hydrogen generated.

Experimental.—When the flask A has been filled as explained above, insert the rubber cork so that the thread holding the HCl tube, keeps it upright, and no acid runs over.

Next, by blowing at *d* and opening clip *k*, let the water fill the tube which enters C.

When the water is running over, close the clip *k*, and then join the flask A to the rest of the apparatus by rubber tubing at *d*. If there is no leakage, there should be no flow of water through C (beyond a few drops at first) when the clip *k* is opened.

Finally, pour in water while clip *k* remains open, until the level in C is the same as that in B. This ensures that the pressure in B is atmospheric.

Now close the clip *k*. Pour out the water in C, and replace the cylinder. Tilt a little acid over in A, and simultaneously open clip *k*. As the action slows down, tilt more acid over, till finally all the acid has been used and the zinc has entirely dissolved. When this is the case, adjust the cylinder C so that the levels in B and C are nearly the same. Allow to cool for five minutes, and then adjust the levels of B and C exactly and shut the clip *k*.

Pour the water collected, into a measuring cylinder and find its volume. This volume equals the volume of hydrogen evolved. Correct it for temperature and pressure, and so reduce its value to N.T.P. (see p. 186).

Knowing that 1000 c.cms. of hydrogen at N.T.P. weigh 0·0897 gm., calculate the weight of hydrogen collected.

From this, calculate the weight of zinc necessary to give 1 gm. of hydrogen. This number is the chemical equivalent of zinc.

Exp. 2. To Find the Equivalent of Tin by Converting it to SnO_2.

When metallic tin is treated with strong nitric acid it becomes oxidised to stannic acid, which on heating loses water, passing to stannic oxide.

(i) $$Sn + 2HNO_3 = H_2SnO_3 + N_2O_3.$$
(ii) $$H_2SnO_3 = H_2O + SnO_2.$$

The equivalent of oxygen is eight, hence by calculating the weight of tin combined with 8 gms. of oxygen the equivalent of tin is obtained.

Experimental.—Place about 1 gm. of granulated tin (accu-

rately weighed) in a small, weighed evaporating dish. Just cover it with distilled water, then add an equal volume of concentrated nitric acid and cover immediately with an inverted funnel to prevent spirting of the liquid. If all the tin has not disappeared when the vigorous reaction ceases, add a little more nitric acid. Finally, when the tin has quite dissolved, leaving only a white powder, remove the funnel, wash any splashes into the dish, and evaporate to dryness on the water-bath. Then ignite gently over a Bunsen flame, and weigh the resulting dry SnO_2 when cold. The increase in weight equals the weight of oxygen combined with the tin.

*Exp. 3. To Find the Equivalent of Copper by Replacement with Zinc.

When metallic zinc or iron is placed in copper sulphate solution, the copper is displaced by the metal and deposited in a powdery form. The weight of copper deposited is equivalent to the weight of zinc or iron dissolved.

Experimental.—Weigh accurately about 1 gm. of zinc foil. Place it in a beaker, and pour on to it about 100 c.cms. of 10 per cent. copper sulphate solution. Warm gently for a quarter of an hour and stir. When all the zinc is dissolved, pour the warm liquid through a weighed filter-paper, and so collect the precipitated copper.

Wash it well with warm water, and then place filter and funnel in the steam-oven to dry. When the filter and contents are quite dry, let them cool in the desiccator, and then weigh.

The equivalent of zinc is 32·5. Calculate from the results of this experiment, the weight of copper which this zinc would replace.

Exercise.—Find by the same method as above the equivalent weight of copper, using about 1 gm. of iron filings.

The equivalent of iron is 28·0.

Exp. 4. To Find the Equivalent of Copper and of Oxygen by Electrolysis.

Faraday's Law.—When the same current passes through two or more electrolytes, the quantities of the elements liberated, are proportional to their chemical equivalents.

Experimental.—The apparatus needed is shown in Fig. 32. It consists of a copper sulphate voltameter, A, and a water voltameter, B.

The copper voltameter consists of a beaker large enough to accommodate two copper plates, each having an area of 3×6 ins.

The beaker is filled with dilute copper sulphate solution (10 per cent.), and a binding screw is fixed to the top edge of each plate, to serve as a terminal.

The water voltameter consists of a large funnel, inverted

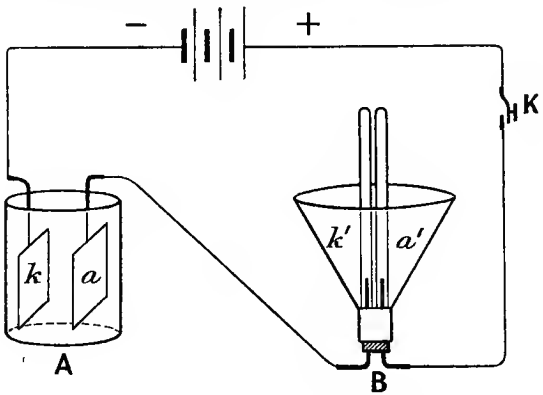

FIG. 32.

and corked with a rubber cork, and is filled with dilute sulphuric acid (1 : 4).

Through the cork, two platinum terminals pass, and over each terminal, is placed a graduated glass tube capable of holding about 20 c.cms. Both tubes must be filled with dilute sulphuric acid before being inverted and placed in position.

These two voltameters are connected in series, so that the same current traverses both, and the current itself is taken from a battery of three Bunsen cells, or accumulators, arranged in series. A key, K, may be included in the circuit. If the current is allowed to pass for about twenty minutes round this circuit, metallic copper will be deposited upon *k* in the copper voltameter, hydrogen will be liberated

in the graduated tube k', and oxygen will be liberated in the graduated tube a'.

First weigh the kathode plate while dry. Next place it in the voltameter, and arrange the rest of the apparatus as in the figure. Now close the circuit by means of the key, and allow the current to pass until about 20 c.cms. of hydrogen has collected in k'. Then break the circuit and remove the copper kathode, wash it with distilled water, and finally with alcohol, then put it in the steam-oven to dry, and when dry weigh it.

The gain in weight represents the weight of copper deposited.

Next, measure the volume of hydrogen evolved in k', while the water-level is the same inside and outside the tube.

This volume must be corrected to 0° C. and 76 cms. pressure, and it should be remembered that the pressure in the tube is that of the atmosphere, less the tension of aqueous vapour at the temperature of the experiment (see page 187).

From the corrected volume, the weight of hydrogen can be calculated on the basis that 1 c.cm. of the gas = 0·00009 gm.

When this has been done, measure the oxygen in the same manner, and calculate its weight, being given that 1 c.cm. of oxygen = 0·00143 gm.

Now, on the basis of hydrogen = 1, calculate the equivalent weights of oxygen and copper which have been liberated.

CHAPTER V

SULPHIDES—COMPOUNDS OF THE ELEMENTS WITH SULPHUR

ALL the metals, in a finely divided condition, combine with sulphur under the influence of heat, forming sulphides. In Exp. 8, page 24, iron sulphide was prepared in this way.

Sulphur is a crystalline yellow solid, which melts at 119° C. to an amber liquid. On further raising the temperature, it becomes darker in colour and viscous. It subsequently becomes more fluid again, and finally boils at 446° C.

*Exp. 1. To Prepare and Collect Hydrogen Sulphide.

The fœtid-smelling gas obtained in Exp. 9, page 25, by treating iron sulphide with dilute hydrochloric acid, may be prepared in quantity and collected as follows:—

In the 8-oz. flask, fitted as in Fig. 33, place about 10 gms. of iron sulphide. Just cover it with water, and replace the stopper. The gas should be collected by downward displacement, as it is rather soluble in cold water, and is slightly denser than air. When a jar has been placed under the delivery-tube, pour a few drops of strong hydrochloric acid down the thistle-funnel. The effervescence which follows, indicates that gas is being evolved and expelled from the generating flask. The gas-jar is full when a piece of lead acetate paper, held at its mouth, turns black.

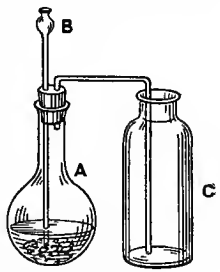

FIG. 33.—(*From O'Shea's "Chemistry for Coal-Mining Students."*)

When the jar is full, close it with a well-greased plate, and fill two more jars in the same way. Then put the flask into the fume-cupboard.

Note that the gas (hydrogen sulphide) is colourless, and possessed of a most disagreeable odour.

Conduct the following experiments with the three jars, having first obtained a trough half-filled with water.

Jar 1.—Remove the glass plate and bring a lighted taper to the mouth. The gas burns with a blue flame, but the taper on being pushed right into the jar is extinguished. Hence, H_2S burns with a blue flame but does not support combustion. Note the deposit of sulphur on the jar, which has been produced during the burning—

$$H_2S + O = H_2O + S.$$

Jar 2.—Introduce about 20 c.cms. of cold water into this jar. Put on the glass cover tightly, and while holding it in place shake the water from end to end. Now invert the jar under water and then remove the cover. The rise of water which follows will give some idea how soluble the gas is in water.

Jar 3.—Introduce into the jar about 20 c.cms. of caustic soda, and shake well with the glass cover firmly in position. Invert under water and then remove the plate. Compare the solubility of the gas in caustic soda with its solubility in water. Finally, take a piece of bright copper, a silver coin, and a piece of bright lead, and place each in turn under the delivery tube in the fume-cupboard. Note how each bright metal becomes rapidly tarnished.

Exp. 2. To Prepare a Sulphide by Reducing a Sulphate.

Take about 10 gms. of the mineral, heavy-spar (barium sulphate), and intimately mix it, in powder form, with about 2 gms. of powdered wood charcoal. Place the mixture in a porcelain crucible, cover with a thin layer of charcoal, and put on the crucible lid.

Heat the contents by placing the crucible on a pipeclay triangle over a small Bunsen flame. After a few moments increase the heat, and ultimately keep the bottom of the crucible bright red, for a quarter of an hour; then remove the burner and allow the contents to cool. When cold, transfer the mixture to a small evaporating dish, treat it with about

20 c.cms. of water and stir well. The barium sulphide which has been formed, dissolves in water, while the charcoal remaining, and unchanged $BaSO_4$, are insoluble. Filter, and collect the filtrate in a small beaker. To prove that the solution contains barium sulphide, pour a few drops into a test-tube and add to it a little dilute hydrochloric acid. Effervescence takes place, and a gas is evolved with fœtid odour, which blackens a lead acetate paper. This gas is H_2S.

The reactions here are :—

Reduction—

(*a*) $BaSO_4 + 4C = BaS + 4CO.$
Barium sulphate. Barium sulphide. Carbon monoxide.

(*b*) $BaS + 2HCl = BaCl_2 + H_2S.$
Barium chloride. Hydrogen sulphide.

***Exp. 3. To Prepare Metallic Sulphides (Precipitation).**

When H_2S is passed through solutions containing metals, a precipitate is generally produced. The colours of these sulphide precipitates are so characteristic that they are used to identify certain metals, in analysis.

A solution obtained by passing H_2S into ammonia water (ammonium hydroxide), may be used more conveniently. Such a solution is known as ammonium sulphide.

Take solutions of the salts named; place each in a clean test-tube, and add ammonium sulphide till no further ppt. is produced.

The salts to be used, dissolved in distilled water, are :—

Lead nitrate, copper sulphate, antimony chloride, zinc sulphate, ferrous sulphate, manganous sulphate.

In each case allow the ppt. to settle at the bottom of the tube, pour off the upper liquid, and then wash the ppt. once by decantation with distilled water (see p. 7).

The washed ppts. are next treated with a few c.cms. of 5 per cent. hydrochloric acid, and warmed gently. Note those ppts. which dissolve in the acid and those which do not, and also make a note of the colour of each sulphide precipitated.

*Exp. 4. To Prepare a Metal (Lead) from its Sulphide.

The names of the important sulphides which occur as minerals are given on page 170.

Galena is a fairly pure form of lead sulphide, and is the mineral from which metallic lead is prepared.

Place 25 gms. of powdered galena in a small fireclay crucible, and mix it thoroughly with, 20 gms. of fusion mixture, 6 gms. of iron filings, and ½ gm. of powdered wood charcoal. Then cover the mixture with a thin layer of fusion mixture, put on the lid, and place the crucible in a muffle furnace which has been raised to a red heat.

The crucible must remain in the furnace for half an hour. At the end of this time the reaction will be complete, and the contents are removed from the furnace by tongs and poured quickly into an iron mould.

When the residue is cool, turn it out of the mould, and detach any slag from the lead button by hammering.

Finally, weigh the metal obtained, and calculate the percentage yield of lead from the galena used.

Exp. 5. To Prepare Mercury from Cinnabar.

Cinnabar is mercury sulphide, the most important and common ore of mercury, and when strongly heated with quicklime it decomposes and gives up all its mercury according to the following equation :—

$$HgS = Hg + S.$$

The lime absorbs the sulphur and facilitates the decomposition.

Method.—Take a hard glass tube, 30 cms. long, sealed at one end. Introduce into this tube a mixture of 5 gms. of powdered cinnabar, with enough powdered quicklime to one-third fill the tube. The mixing should be done in a mortar, and the transfer to the tube accomplished by a piece of black glazed paper. Wash out the mortar with more quicklime, and transfer this also to the tube, which should now be filled as indicated here (Fig. 34).

From A to B is the mixture of cinnabar and quicklime. From B to C is dry quicklime in small lumps.

Now draw out the tube at D so as to obtain a bend of narrow tube, thus (Fig. 35). Clamp the tube in a retort-stand by means of copper wire *k k'*, and let the open and drawn-out end *just* dip under cold water in a small beaker. Tap the tube to make a small air channel.

FIG. 34.

Gently heat[1] the front end of the tube which contains the lime only, and gradually increase the heat to bright redness. Then work the flame back towards the sulphide mixture, keeping the portion BC red hot all the while. Ultimately the whole tube may be raised to a bright red heat, and kept at this temperature for a moment or two.

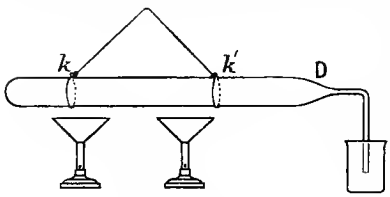

FIG. 35.

Now remove the burner, raise the exit tube from the water, and, while the tube is still hot, cut across it with a file at *d*, and tap down any mercury globules which may have collected in the narrow tube.

Rotate the beaker so that the mercury joins up to form one globule, and then pour off the water. Transfer the mercury to a weighed watch-glass, and remove as much water as possible by repeatedly dabbing with filter-paper.

Finally, weigh the dried metal, and calculate from the yield, the percentage of mercury obtained from the specimen of cinnabar.

[1] The heating may be done by using two Bunsen burners with flattened flames (see Fig. 35).

CHAPTER VI

CHLORINE AND ITS COMPOUNDS

THE importance of chlorine is realised when we remember that it is a constituent of three most valuable substances, in common and everyday use—namely, common salt, hydrochloric acid, and "chloride of lime" or bleaching powder.

***Exp. 1. To Prepare Hydrochloric Acid Gas from Salt.**

Weigh out about 10 gms. of common salt (sodium chloride) and place it in an 8-oz. conical flask, fitted with thistle-funnel and delivery-tube as shown in Fig. 36. Cork the flask and make the funnel reach the salt. Prepare some sulphuric acid solution by gradually adding 16 gms. concentrated H_2SO_4 to 10 c.cms of water. The water is in a boiling-tube, and after each addition of sulphuric acid, the tube is shaken and held under the tap so that cold water falls on the outside and keeps it cool. When the mixing is completed, transfer the contents to the flask containing the salt. Place a gas-jar under the

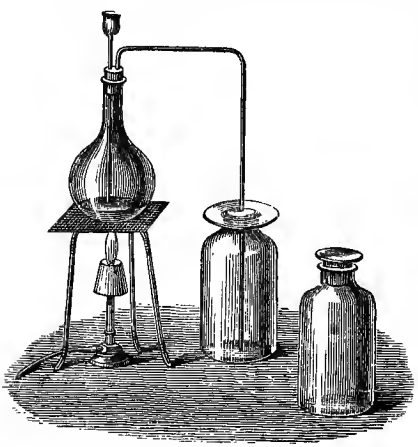

FIG. 36.—(*From Thorpe's "Qualitative Chemical Analysis."*)

delivery-tube, and if the gas is not evolved quickly enough, warm the flask gently. When the jar is full, a lighted match held at the mouth of the jar will be extinguished. Collect three jars full, and close each one with a well greased glass plate. Now remove the cork from the flask, transfer flask and tripod to the fume-cupboard, and continue heating until no more fumes of HCl are evolved. While this is going on, test the three jars of hydrochloric acid gas as follows :—

Jar 1.—Remove the glass plate and notice how the gas fumes in air. Put into the fumes a piece of blue litmus paper, and observe the reddening which takes place. Then bring up to the jar a lighted taper. It is extinguished on entering the jar, and further, the gas does not burn.

Jar 2.—Take an empty gas-jar and rinse round it a few c.cms. of strong ammonia solution. Place it on the bench, mouth upwards, and then bring a jar of HCl gas (with glass plate still on), in an inverted position, over the jar of ammonia. Remove the glass plate which separates the two jars, and observe the immediate formation of dense white fumes of ammonium chloride. These are formed by the union of the two gases—

$$\underset{\text{Ammonia.}}{NH_3} + \underset{\text{Hydrochloric acid.}}{HCl} = \underset{\text{Ammonium chloride.}}{NH_4Cl.}$$

Jar 3.—Invert this jar (with glass plate on) over cold water in a trough, and then when the mouth is under water, remove the glass plate. From the manner in which the water rises, observe that the gas dissolves readily in water. The solution of the gas in water is known as hydrochloric acid, or commercially as " spirits of salt."

***Exp. 2. To Prepare Crystals of Acid Sodium Sulphate.**

When fumes of HCl have ceased to be expelled from the flask in the fume-cupboard, the reaction between sulphuric acid and salt is complete, and the thick liquid contains acid sodium sulphate. Remove the flask, and pour in gradually enough distilled water to just dissolve any crystals present. Then pour the contents of the flask into an evaporating dish. Evaporate the liquor until crystals form on its surface, and

then remove and cool. When quite cold, a good crop of acid "salt cake" crystals will be present. Pour off the mother-liquor and let the salt drain.

Recrystallise the acid sodium sulphate by redissolving in just sufficient hot water and evaporating as before. Remove the well drained crystals to a pad of filter-paper, and place in the steam-oven. When quite dry weigh them, and note down the yield of "salt cake" got from the sodium chloride.

These two experiments are a copy on a small scale of the process for manufacturing hydrochloric acid, and "salt cake" or sodium sulphate.

In the manufacture, the gas is collected in water to form hydrochloric acid, and the mixture of salt and sulphuric acid is heated to a higher temperature, so that sodium sulphate or real *salt cake* is obtained.

In our experiment the reaction was :—

$$NaCl + H_2SO_4 = \underset{\text{Acid sodium sulphate.}}{NaHSO_4} + HCl.$$

On the manufacturing scale the next and final stage is :—

$$NaHSO_4 + NaCl = \underset{\text{Sodium sulphate.}}{Na_2SO_4} + HCl.$$

*Exp. 3. To Prepare Chlorine Gas from Hydrochloric Acid.

Place 10 gms. of granulated manganese dioxide in an 8-oz. conical flask, fitted with thistle-funnel and delivery-tube as shown in Fig. 37. A Woulffe's bottle containing cold water must be used to wash the chlorine gas free from any hydrochloric acid which passes over. Measure into a beaker 80 c.cms. of concentrated hydrochloric acid, add 25 c.cms. of water, and pour the acid mixture into the flask, through the funnel. Put a *small flame* under the flask, and after allowing the air to be displaced, collect three jars of the greenish-yellow gas evolved, by downward displacement, and cover each with a greased plate.

The jar is full when a lighted match, held to its mouth, is extinguished.

When three jars have been collected, place a fourth jar under the delivery-tube, so that the latter nearly touches the bottom of the jar, and then drop in small pieces of soda-lime so as to fill a depth equal to one-fourth. This will serve as a trap to catch the chlorine gas, preventing it from getting into the air, and at the same time, it will convert some of the soda-lime into bleaching powder.

Test the three jars of gas as follows:—

Jar 1.—We have already observed that the gas extinguishes a lighted match. Observe its greenish-yellow colour, and also its characteristic pungent odour. Plunge into the jar a burning taper, and note that it continues to burn feebly in the chlorine, emitting much soot. The hydrogen in the wax combines with the chlorine to form HCl, while the carbon in the wax is deposited as soot.

When the taper has burnt for a few seconds in the jar, remove it, and bring into the jar, a rod moistened with strong ammonia solution. The dense white fumes formed, show the presence of hydrochloric acid.

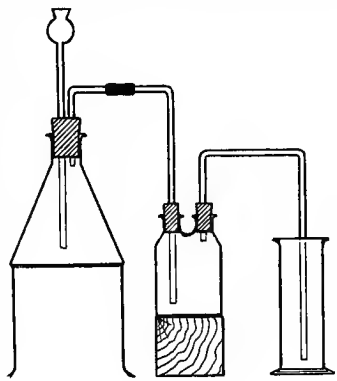

FIG. 37.

Jar 2.—Warm a few c.cms. of turpentine in a test-tube, by immersing the tube in a beaker of hot water. When the turpentine is warm, push a filter-paper into the tube, so that it soaks up and becomes saturated with the oil. Now drop the soaked filter-paper into the second jar of chlorine gas, and note how it smoulders and finally burns with a very smoky flame. The hydrogen in the oil is combining with the chlorine to form HCl, and the carbon is deposited as soot. Prove that the jar now contains HCl by means of a glass rod moistened with ammonia solution.

Jar 3.—Pour into this jar about 20 c.cms. of water, and shake up well. Notice that the yellow gas dissolves in the water, imparting to the solution a yellow colour. This is

known as chlorine water. Put into the chlorine water a piece of turkey-red cloth, and shake round well. In a few minutes the colour will be completely discharged, on account of the bleaching action of moist chlorine.

*Exp. 3a. To Decolourise a Piece of Dyed Cloth by Bleaching Powder Solution.

Remove the flame now from the chlorine generating-flask. Disconnect the flask, and place it in the fume-cupboard. Take the jar containing the soda-lime, which will now be partly converted to bleaching powder. Pour it into a trough half-full of water, and stir well so as to make most of the solid dissolve. This solution represents a bleaching-bath such as is used on a commercial scale. Place by the side of this a large beaker containing dilute sulphuric acid. This represents the acid-bath.

Now bleach a piece of red or blue cloth by immersing it, first in the bleaching-bath and then in the acid-bath. After a few seconds in each bath, repeat the process until the colour has been completely removed. It is necessary to place the fabric in the acid, so that chlorine may be liberated from the bleaching powder solution.

The preparation of chlorine by oxidising hydrochloric acid with manganese dioxide, is represented thus:—

$$MnO_2 + 4HCl = MnCl_2 + 2H_2O + Cl_2.$$

Note.—The preparation of common salt is described in Exp. 5, p. 40.

*Exp. 4. Action of Peroxides upon Hydrochloric Acid.

The peroxides were referred to on p. 44, and in the preparation of chlorine, the action of manganese dioxide upon hydrochloric acid was studied.

Place in three test-tubes small quantities of:—

(1) Red lead; (2) lead peroxide; (3) barium peroxide.

Take each tube in turn and add a few drops of concentrated HCl, and warm gently.

Observe in each case the green-yellow colour of the evolved gas, and note that it smells of chlorine and bleaches a piece of moist litmus paper.

The composition of these three peroxides is as follows:—
Red lead = Pb_3O_4; lead peroxide = PbO_2; barium peroxide = BaO_2.

Exp. 5. Silver Chloride.

To a few drops of silver nitrate solution in a test-tube, add sodium chloride solution, until no further white ppt. results. Let the ppt. of silver chloride settle, and then pour off the clear liquid. Add distilled water (half a tubeful), shake up, and divide the suspended ppt. into two equal portions.

Add to one, a few drops of ammonia water, shake up, and note how easily the silver chloride dissolves. Add to the second portion a few drops of strong nitric acid and shake up. The ppt. does not dissolve. Silver chloride, therefore, dissolves in AmOH, but not in nitric acid.

*Exp. 5a. Lead Chloride.

To a few c.cms. of lead nitrate solution in a test-tube, add sodium chloride solution, until no further white ppt. results. Allow the ppt. to settle, and then pour off the clear liquid. Add half a tube of distilled water, and boil. The lead chloride completely dissolves when the water boils, and on cooling the tube, fine glistening crystals of the salt are deposited.

The above reactions are represented thus:—

(1) $$\underset{\text{Silver nitrate.}}{AgNO_3} + \underset{\text{Sodium chloride.}}{NaCl} = \underset{\text{Silver chloride.}}{AgCl} + \underset{\text{Sodium nitrate.}}{NaNO_3}.$$

(2) $$\underset{\text{Lead nitrate.}}{Pb(NO_3)_2} + 2\underset{\text{Sodium chloride.}}{NaCl} = \underset{\text{Lead chloride.}}{PbCl_2} + 2\underset{\text{Sodium nitrate.}}{NaNO_3}.$$

Exp. 6. Bromine.

Mix together equal quantities of powdered potassium bromide and manganese dioxide, using a sufficient quantity to fill the boiling-tube used, to a depth of one inch. Arrange a clamp so that the boiling-tube, fitted with a cork and right-angled tube, may be fixed in position, as shown in Fig. 38, together with a test-tube receiver, dipping in a beaker of

cold water. Now add concentrated H_2SO_4 to the contents of the boiling-tube, with shaking, so as to form a thick paste, and then replace the cork. A deep-red gas will be evolved, filling the tube, and some will pass into the delivery-tube, where it will condense in red drops. Warm gently and expel the bromine which is formed, so that it condenses at the bottom of the cooled receiving-tube. When no more bromine is evolved, remove the flame and examine the nature of the liquid at the bottom of the test-tube. It is heavy, deep red in colour, and the smell (observe with care) resembles that of chlorine, but is much more irritating to the throat. It boils at 59° C.

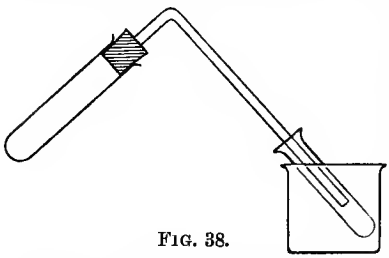

FIG. 38.

(*a*) Place in the red vapour a piece of starch-paper, and note that it is turned orange in colour.

(*b*) Pour a few drops of the heavy liquid into another test-tube. Add a few c.cms. of distilled water, and shake well. The bromine completely dissolves in the water, yielding a red solution known as bromine water.

(*c*) Pour into the tube, which contains the rest of the bromine, a few c.cms. of carbon bisulphide, and shake. The bromine again dissolves completely, giving a bright red solution.

Equation—

$$\underset{\text{Potassium bromide.}}{2KBr} + 2H_2SO_4 + \underset{\text{Manganese dioxide.}}{MnO_2} =$$

$$\underset{\text{Potassium sulphate.}}{K_2SO_4} + \underset{\text{Manganese sulphate.}}{MnSO_4} + 2H_2O + Br_2.$$

CHAPTER VII

CARBON DIOXIDE AND CARBONATES—CARBIDES

THE most widely distributed and most important carbonate among those classed with the natural minerals (table, p. 170) is calcium carbonate, which as marble and limestone occurs in very large quantities. The most characteristic property of these bodies is that on being strongly heated they decompose into an oxide of the metal and a colourless, heavy gas, namely, carbon dioxide.

This same gas is formed by the combustion of carbon and carbonaceous substances, like coal and wood, and it was formed in this manner in Exp. 8, p. 35, by the combustion of carbon in oxygen.

*Exp. 1. To Prepare Carbon Dioxide by Heating a Carbonate.

Place in a hard glass tube enough copper carbonate to fill the tube to a depth of 2 to 3 inches, and fit it with a single-bored cork and a delivery-tube bent at an angle of about 80°. Clamp the tube on a retort-stand, so that the delivery-tube points vertically downwards, and into a test-tube, in which the gas may be collected. Now gently heat the tube containing the copper carbonate, and when the glass has become well warmed, heat strongly. The carbonate darkens in colour, and ultimately becomes black, while gas is evolved and collected in the second test-tube by downward displacement. As soon as a lighted match, placed at the mouth of the collecting tube, is extinguished, the tube is full of carbon dioxide. Cease heating and remove the test-tube of gas, covering the open end with your thumb. Introduce into the gas collected, a lighted taper or match, and note that it is immediately extinguished.

Collect a second tube of the gas by again applying heat, remove when full, and close with the thumb as before. Pour into this tubeful a few c.cms. of clear lime-water, and shake up. The lime-water becomes milky, because calcium carbonate is precipitated.

Equation—

$$\underset{\text{Calcium hydroxide.}}{Ca(OH)_2} + CO_2 = \underset{\text{Calcium carbonate.}}{CaCO_3} + H_2O.$$

*Exp. 2. Changes produced in Calcium Carbonate by Strongly Heating.

Calcium carbonate loses CO_2 when strongly heated, and is converted into quicklime (calcium oxide).

Equation—

$$CaCO_3 = CaO + CO_2.$$

Weigh into a clean porcelain crucible 1 gm. of dry calcium carbonate. Place the crucible upon a pipeclay triangle fixed on a tripod (see Fig. 16), and heat the crucible by a Bunsen flame, gently at first, and finally to bright redness. The lid should not be on, so that the gaseous CO_2 may escape readily. When the contents have been heated to bright redness for twenty minutes, remove the burner. Allow the crucible to cool somewhat, and while still hot, place it in a desiccator to finish cooling. Weigh when quite cold, and calculate the percentage loss which the carbonate undergoes when it decomposes into quicklime and carbon dioxide.

Divide the residual calcium oxide into two parts.

(*a*) Pour on to one part a few drops of dilute hydrochloric acid. Little or no effervescence takes place.

Now pour a few drops of the acid on to an equal quantity of the original carbonate, and note the vigorous effervescence.

(*b*) Add a little cold distilled water to the other part, and note the intense heat developed during this SLAKING process. Add more water, and into the liquid dip a red litmus paper. The paper is turned blue, showing that the slaked lime is alkaline.

If a piece of red litmus be dipped into moistened chalk (calcium carbonate), it is not turned blue.

***Exp. 3. To Prepare Carbon Dioxide in Quantity.**

Fit up the flask and delivery-tube as shown in Fig. 33. Place between 15 and 20 gms. of marble, broken in small pieces, in the generating flask, carefully. Replace the cork, and pour in just enough water to reach the lower end of the thistle-funnel tube. Place a gas-jar under the delivery-tube, and then pour into the flask, a little at a time, 60 c.cms. of concentrated hydrochloric acid. Vigorous effervescence shows that gas is being evolved, and the gas-jar is full when a lighted match, placed at the mouth, is extinguished. Remove this jar, and cover with a greased glass plate. Fill two more jars in the same way.

Now let the delivery-tube dip into clear lime-water (about 25 to 30 c.cms.) contained in a small beaker, and let the evolution of gas continue, while the three jars collected are used as follows :—

Jar 1.—The action of the gas on lime-water, and on a burning match, has been noted. Note further that the gas is colourless, but possesses an acid smell and taste. Pour into the jar a few c.cms. of blue litmus solution, and observe the change in colour to red. This indicates that the gas is acid and soluble in water.

Jar 2.—Fix a few inches of magnesium ribbon in crucible tongs, light it, and plunge it into the jar of CO_2. It continues to burn, and a deposit of carbon, as well as white oxide of magnesium, forms inside the jar. The burning magnesium is able to decompose the CO_2 into carbon and oxygen, and so continues burning in the oxygen formed.

Equation—

$$CO_2 = C + O_2.$$

Jar 3.—Pour into this jar a few c.cms. of sodium hydroxide solution, and quickly close the opening with a greased plate. Shake round the liquid and then invert the jar, with its mouth under water, in a large beaker or trough. Now remove the glass plate; notice that this is somewhat difficult, an indication that the gas has been dissolved inside, thus creating a partial vacuum. When the plate is removed, the water rises quickly and practically fills the jar. This proves that sodium hydroxide solution is a good absorbent for CO_2.

Equation—

$$\underset{\text{Sodium hydroxide.}}{2NaOH} + CO_2 = \underset{\text{Sodium carbonate.}}{Na_2CO_3} + H_2O.$$

Now turn attention to the beaker of lime-water into which the CO_2 has been passing. At first the usual turbidity is produced, but after allowing the gas to pass for some time, the liquid again becomes clear. This is due to the fact that excess of CO_2, converts the calcium carbonate into calcium bicarbonate, which is soluble in water.

Equation—

$$CaCO_3 + CO_2 + H_2O = Ca(HCO_3)_2.$$

Divide this solution into two equal parts, and boil one part for two minutes. The turbidity reappears, owing to the expulsion of CO_2 by the heat, and $CaCO_3$ is reprecipitated.

Equation—

$$Ca(HCO_3)_2 = CaCO_3 + HO_2 + CO_2.$$

Now shake up in a small stoppered bottle, first, the unboiled lime-water, and then the one which has been boiled, each with sufficient soap solution to produce a lather.

Notice that the amount of soap solution required to give a lather with the unboiled sample is much larger than that required by the one which has been boiled. The former is a "hard" water, and some of it has been "softened" by the process of boiling. The hardness of water, *i.e.* that property which prevents it from lathering freely with soap, is due to the salts in solution. Table XX. p. 184, gives the substances usually present in natural water, and it is to *some* of these that hardness is due, most notably, bicarbonates of lime and magnesia, together with the sulphates and chlorides of lime and magnesia. These salts may be removed completely, and the water purified and softened by the process of distillation.

*Exp. 4. To Purify River or Sea Water by Distillation, and to Compare the Hardness of Different Waters.

Clamp a small retort (about 8 or 10 oz.) as shown in Fig. 39. Let the neck slope downwards and dip into a clean

small flask, which is kept cool by immersion in a trough of cold water. Pour into the retort some river or sea water until nearly half-full. Drop in a piece of porous pot to prevent "bumping" of the liquid, and then place the stopper in the tubule. The retort may now be heated by means of a flame placed beneath it. The first few c.cms. which collect in the receiver should be thrown away, in case the retort neck or receiving flask was not quite clean. When half the water in the retort has distilled over, remove the burner and stop the process.

While the distillation is proceeding, test the original water in the following ways, using in each case a few c.cms. of the water in a clean test-tube:—

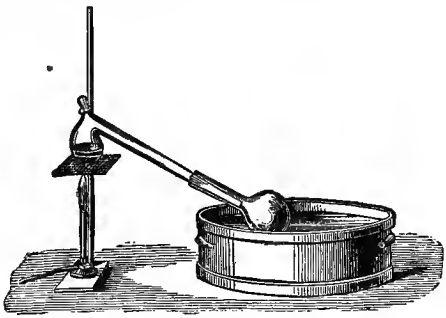

FIG. 39.—(*From Thorpe's "Qualitative Chemical Analysis."*)

(*a*) Add a few drops of dilute nitric acid and then a few drops of silver nitrate solution. A white ppt. indicates *chloride.*

(*b*) Acidify another portion with a few drops of dilute hydrochloric acid, and add barium chloride solution. A white ppt. indicates the presence of *sulphate.*

(*c*) Add a few drops of dilute ammonia, and then ammonium oxalate. A white ppt. indicates *calcium.*

Repeat these tests with three separate portions of the water which has distilled over, to prove the absence of these impurities. In each case the water should remain clear, giving no ppt.

Evaporate 50 c.cms. of the distilled water to complete dryness on a water-bath, and compare the residue with that obtained when 50 c.cms. of the water in the retort is evaporated to dryness.

*Exp. 4a. To Compare the Hardness of Distilled Water, Tap Water, and Sea or River Water.

Fill a burette with soap solution, and measure into a clean stoppered bottle 50 c.cms. of distilled water. Run in from the burette, one drop at a time, soap solution, until on stoppering and shaking up, a lather is obtained which remains permanent for half a minute. Note the volume of soap solution used.

Rinse the bottle well with distilled water, and use next, 50 c.cms. of tap water. The soap solution may be run in fairly rapidly at first, and with greater care when a lather begins to form on shaking. Note the volume used when a permanent lather is obtained.

Well clean the bottle again, with distilled water, and use for the third test 50 c.cms. of sea or river water, and measure the volume of soap solution required here. How do these three waters compare as regards "hardness"?

*Exp. 5. To Soften Hard Water.

Take 50 c.cms. of the sea or river water, and boil it in a small beaker for three minutes. Allow it to cool, and then pour it into the stoppered shaking-bottle, and find out how much soap solution is required for a permanent lather. It will be less in quantity than that required by the original unboiled water. Part of the hardness, namely, that due to bicarbonates, has been removed by the boiling, and such hardness is called "temporary hardness."

Take another 50 c.cms. of the original water, boil it for two minutes in a small beaker to remove the temporary hardness, and then, while still hot, add a few c.cms. of sodium carbonate (washing soda) solution, and pour the whole into the stoppered shaking bottle. Run in soap solution carefully, and measure the amount required for a permanent lather.

The amount used now is very small, for both "temporary" and "permanent" hardness have been removed.

"Permanent hardness," due to sulphates and chlorides of lime and magnesia, cannot be removed by boiling, but is got rid of, by the addition of washing soda.

Exp. 6. To Prepare Crystals of Washing Soda ($Na_2CO_3,10H_2O$).

Pour into a small beaker 50 c.cms. of 2N (double normal) solution of sodium hydroxide. Pass a steady stream of CO_2 into this solution from the generating flask used previously, or a Kipp, and allow the gas to pass until the solution in the beaker ceases to turn red litmus blue. This is a sign that all the alkali has been converted into Na_2CO_3. Remove the beaker, pour the contents into a small evaporating dish, wash the beaker once with a few c.cms. of distilled water, and add the washings to the dish. Now evaporate the solution over a small flame till crystallisation commences, and then place it on one side to cool and crystallise. In order to get small crystals which can be dried easily, stir the contents while cooling. The cooling may be hastened by standing the dish in cold water. When quite cold, drain off any water remaining, transfer the crystals to filter-paper, well press them, and when dry, put them on a small filter-paper and weigh them. The yield of washing soda crystals should be about 12 gms.

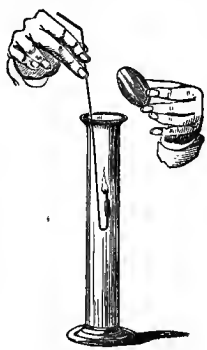

FIG. 40.—(*From Miller's "Inorganic Chemistry."*)

*Exp. 7. To Prove that Carbon Dioxide is formed in the Processes of Breathing and Combustion.

(*a*) Place a few c.cms. of clear lime-water in a small beaker, and taking a clean glass tube, blow through the tube so that the exhaled breath passes through the lime-water. After a short time it becomes turbid, a proof that CO_2 is breathed out from the lungs.

(*b*) Fix a small piece of candle on to a deflagrating spoon, so that it can be lowered into a gas-jar. Light the candle, and, while it is burning, lower it into a clean gas-jar (see Fig. 40), and let it burn out. When the candle has used up all the oxygen in the jar, combustion is no longer possible,

and the candle is extinguished. Now remove the deflagrating spoon, and pour into the jar a few c.cms. of clear lime-water; put on the cover and shake well. The turbid appearance of the lime-water shows, that CO_2 has been formed by the combustion of the carbon in the candle.

Exp. 8. To Measure the Volume of CO_2 evolved by the Action of Acid on Calcium Carbonate.

For this experiment use the same apparatus as described in Fig. 31. In this case, however, since CO_2 is to be collected in the "Winchester," and since this gas is fairly soluble in water (see p. 65), the water in the large bottle must be previously saturated with CO_2 by adding a few c.cms. of dilute HCl to it, and then a little powdered chalk. When the effervescence has ceased, the water is ready for use. Weigh out accurately, about 1 gm. of powdered pure $CaCO_3$, transfer it carefully to the small flask; fill the small tube with conc. HCl, proceed exactly as in Exp. 1, p. 46, and calculate the volume of gas evolved, at 0° C. and 760 mm. pressure.

If 1 litre of CO_2 gas at N.T.P. weighs 1·973 gms., calculate the weight of gas given off from 1 gm. of $CaCO_3$.

Compare this with the result of Exp. 2, p. 64.

*Exp. 8a. To Estimate the Weight of CO_2 in Iceland Spar.

For this estimation the apparatus shown in Fig. 41 is used. The small flask is fitted with a double-bored rubber cork. Through one hole passes a glass tube, which reaches nearly to the bottom of the flask, and which can be closed by a piece of glass rod and rubber tube at *a b*.

The calcium chloride tube C is to prevent escape of moisture.

Weigh accurately between 0·7 and 1·0 gm. of Iceland spar, place it in the flask, and cover it with distilled water. Fill the small test-tube with strong hydrochloric acid, and fix it by thread passing up the side of the rubber cork, so that the acid remains in the tube without spilling. Make

sure that the cork and tubes are a good tight fit, so that no leakage can take place. When this has been done, and the outside of the flask is quite dry, weigh the complete apparatus.

Next, tilt the apparatus so that a small quantity of acid enters the water, and gradually empty the contents of the acid-tube upon the Iceland spar.

When the effervescence has quite ceased, remove the stopper-rod from *a*, and gently warm the flask on a gauze over a small flame. Do not let it get more than *hot*. Then remove the flame, and, while still hot, fix a piece of rubber tube on to the top end of the calcium chloride drying-tube, and draw a slow stream of air through the vessel for two minutes. This will clear out all the CO_2 from the flask, and the heating will ensure the removal of any CO_2 dissolved in the water. When this has been accomplished, replace the glass tube stopper at *a*, take off the rubber tubing used for aspirating, and let the apparatus cool. When cold, weigh again, and from the loss in weight, due to CO_2 removed, calculate the percentage of CO_2 in Iceland spar.

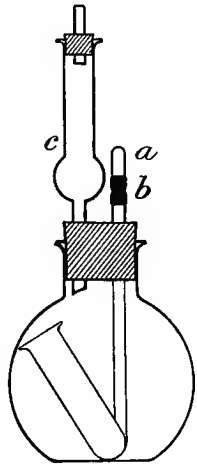

FIG. 41.

Compare the result with that obtained in Exp. 2, p. 64.

Exp. 9. Soap—its Nature and Preparation.

Soap is the sodium salt of certain organic acids (*i.e.* oleic, palmitic, and stearic), and it is made by boiling caustic soda with fat, which contains these acids, combined with glycerin. The following reaction takes place during the process:—

Soda + Glyceryl oleate = Glycerin + Sodium oleate (soap).

This process is called saponification, and the soap itself is precipitated from solution by adding salt. The method is as follows: Tie up 200 gms. of mutton suet in a muslin bag. Place this in a beaker or can of boiling water (500 c.cms.), and by squeezing and melting cause all the fat to ultimately

pass out through the bag into the water. When this is accomplished, remove the residue of tissue and skin which remains in the bag, and add 30 gms. of NaOH dissolved in 100 c.cms. of water. Boil for the space of one hour, and at the end of this time all the fat should be saponified. Now let the solution cool down somewhat, make it up to its original volume, and add 100 c.cms. of saturated salt solution. This precipitates the soap, which must be filtered, washed once with cold water, and then pressed into a cake.

The glycerin, which mixes with water in all proportions, remains in the filtrate, and cannot be recovered in a simple manner.

Carbides.—These substances are hard, metallic-looking compounds, formed by the union of the metals with carbon, the combination being usually brought about by heating the metallic oxide with carbon in an electric furnace. Calcium carbide, the most important, is formed in this manner according to the equation—

$$\underset{\text{Calcium oxide.}}{CaO} + 3C = \underset{\text{Calcium carbide.}}{CaC_2} + \underset{\text{Carbon monoxide.}}{CO}.$$

*Exp. 10. To Investigate the Action of Water on Calcium Carbide (Acetylene).

Fit up a small conical flask (6 oz.) with delivery-tube and thistle-funnel, as indicated in Fig. 42, for collecting a gas over water. Place about 15 gms. of calcium carbide in small pieces, in the flask A, replace the cork, and push the thistle-funnel tube down till it almost touches the bottom of the flask. Now pour in a few c.cms. of water gradually. A vigorous reaction takes place, and gas bubbles rapidly through the water in the trough B. When the air has been cleared out, put a gas-jar filled with water, on the beehive-shelf, and proceed to collect two jars of the acetylene gas which is evolved.

Close each jar with a greased plate, and then collect a third jar thus: Fill the jar one-third full of water, so that two-thirds contains air. Cover with a plate, invert under water, and proceed to fill the jar, so that the one-third of water is expelled. This jar will then contain a mixture of

air and acetylene. Place this jar on the bench, remove the cover, and apply a light. An explosion denotes the fact that the gas is explosive when mixed with air.

Take one of the full jars, remove the cover, and bring a lighted taper to the mouth. The gas burns with an exceedingly luminous and smoky flame, and on thrusting the taper into the jar it is extinguished. Acetylene therefore burns, but does not support combustion. Shake a little lime-water round the jar, and notice its turbid appearance, which proves that CO_2 is formed during the combustion.

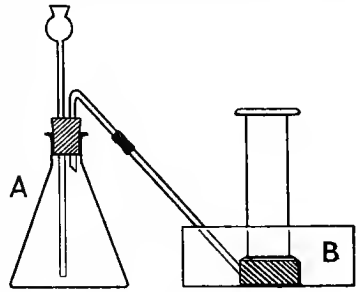

FIG. 42.

Pour into the second jar of acetylene about 10 c.cms. of bromine dissolved in chloroform (5 per cent. solution). Replace the glass plate, and on shaking well, the bromine is completely decolourised.[1] This behaviour is characteristic of those gases known as *unsaturated hydrocarbons*.

Acetylene is produced according to this equation—

$$\underset{\text{Calcium carbide.}}{CaC_2} + 2H_2O = \underset{\text{Calcium hydroxide.}}{Ca(OH)_2} + \underset{\text{Acetylene.}}{C_2H_2}.$$

*Exp. 11. Action of Water on Aluminium Carbide (Methane).

Using the same apparatus as in the last experiment, take about 15 gms. of aluminium carbide and place it in the dry flask. Replace the cork, and then pour small quantities of water through the thistle-funnel, and collect three jars of the gas [2] as before, the third jar being a mixture of air and the gas. Methane, on account of its occurrence in marshy pools, is sometimes termed marsh gas.

[1] If the first jar of gas is not sufficient to decolourise the bromine, pour it into a second jar and shake again.

[2] Gentle warming may be needed to facilitate the evolution of gas.

On applying a light to the jar containing methane and air, an explosion proves its explosive properties.

Apply a light to the second jar inverted, and note that the gas burns with a blue non-luminous flame, and that the taper on being pushed into the jar goes out. Methane therefore burns, but does not support combustion.

Shake up a little clear lime-water in the jar, and so prove that carbon dioxide has been formed by the combustion of methane.

Shake the third jar with 10 c.cms. of bromine dissolved in chloroform, and observe that the colour of the bromine is not discharged. This property belongs to those carbon compounds known as *saturated hydrocarbons*.

Methane has the composition CH_4, and is formed from aluminium carbide according to the following equation :—

$$\underset{\text{Aluminium carbide.}}{Al_4C_3} + 12H_2O = \underset{\text{Methane.}}{3CH_4} + \underset{\text{Aluminium hydroxide.}}{4Al(OH)_3}.$$

Equation representing the combustion of methane—

$$CH_4 + 2O_2 = CO_2 + 2H_2O.$$

Equation representing the combustion of acetylene—

$$2C_2H_2 + 5O_2 = 4CO_2 + 2H_2O.$$

Note.—It has been suggested that the petroleums of Russia and America are due to the action of water, percolating through the earth's crust, upon carbides of the metals which probably exist at a great depth.

The above work indicates the possibility of this being a true explanation.

Petroleum itself is a mixture consisting of various liquid hydrocarbons.

*Exp. 12. To Prepare Carbon Monoxide by Reducing CO_2.

Fit up the apparatus of Fig. 43, which is made up of the following parts: A is a conical flask of 6 oz. to 8 oz. capacity, fitted with a thistle-funnel and right-angled exit tube. About 20 gms. of marble, in small pieces, is placed in the flask, and just covered with water.

A rubber tube at *b* serves to connect on to a combustion tube C (20 cms. long), which is packed with small pieces of

charcoal. The delivery-tube D dips under water in a trough, to which a few c.cms. of caustic soda has been added.

When the apparatus has been arranged as in the figure, commence heating the charcoal-tube C. Gradually raise it to a red heat, and then cause a steady stream of CO_2 to pass through by pouring a little concentrated HCl through the thistle-funnel into A.

Now place a gas-jar over the delivery-tube D, and collect two jars of the escaping gas.

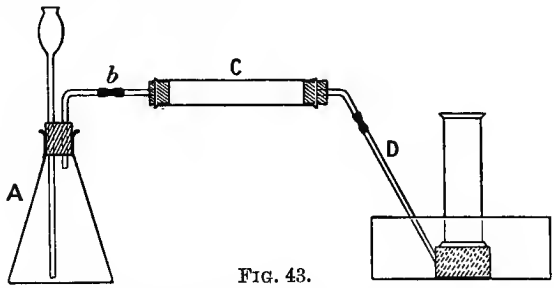

FIG. 43.

When the required gas has been collected, disconnect the apparatus at *b*, and stop heating.

With the two jars of gas, carry out the following experiments:—

Note.—Do not let any of this gas escape into the air of the room, as it is *very poisonous*.

Jar 1.—Remove the glass plate and apply a light. The gas burns with a blue flame. When the flame has ceased to burn, pour in a few c.cms. of lime-water and shake round. A turbidity proves the presence of CO_2.

Jar 2.—Shake up clear lime-water in this jar, and note that it gives no sign of turbidity. Now burn the gas, and then shake the lime-water round. Evidently when carbon monoxide (which is a colourless gas) burns, it is converted into CO_2, which causes clear lime-water to become turbid.

Note.—The reason for placing caustic soda in the trough-water is to absorb any CO_2 which may pass out of the tube. If the stream of gas is *too rapid*, the gas collected will give evidence of CO_2, because some of it may escape absorption.

Equation explaining the conversion of CO_2 into carbon monoxide—

$$CO_2 + C = 2CO.$$

Equation showing how caustic soda absorbs CO_2—

$$2NaOH + CO_2 = Na_2CO_3 + H_2O.$$

Both of these gases are produced when coal or carbonaceous matter burns in air or oxygen. If an excess of carbon be present, then a large percentage of carbon monoxide will be formed, and the characteristic blue flame with which this gas burns is often seen at the top of a fireplace or a furnace chimney.

One gm. of carbon in burning to CO yields 2500 heat units.
" " " " CO_2 " 8080 " "

The heats of combustion of various fuels are given in Tables XI.–XIII. Appendix.

CHAPTER VIII

CARBON AND CARBONACEOUS SUBSTANCES

THE principal carbon-containing bodies which are of use as fuels, are coal, coke, wood, peat, shale, petroleum, and bones.

Carbon burns in air and oxygen, to form carbon monoxide and carbon dioxide. This is the chief chemical reaction taking place in all combustion processes, but in addition, these bodies yield gases such as methane, acetylene, and hydrogen, all capable of burning and serving as heat producers.

Coal is composed principally of carbon, hydrogen, and oxygen, together with smaller amounts of sulphur, nitrogen, ash or mineral matter, and moisture. The composition of various coals is given on p. 181.

The chief products obtained by distilling coal in retorts, are coal gas, coal tar, and ammonia liquor. Other substances produced are CO_2, hydrogen sulphide, carbon bisulphide, and free nitrogen. The coal tar and ammonia liquor are condensed, and subsequently used. The coal gas, after being purified from sulphur gases like H_2S and CS_2, and CO_2, is collected in a gasometer.

The average composition of purified coal gas is: methane, 35 per cent.; hydrogen, 49 per cent.; CO, 7 per cent.; nitrogen, 3 per cent.; CO_2, 1 per cent. Illuminating gases: acetylene, ethylene, and benzene = 5 per cent.

*Exp. 1. To Distil Coal and Collect the Volatile Products.

Take for this purpose the apparatus shown in Fig. 44. A is a hard glass test-tube, two-thirds filled with a weighed quantity of coal, pounded into small lumps. This is clamped with a slight incline, so that any water liberated will not

run back to the hot part of the tube and crack it. The tubes are fitted so that the vapour evolved will pass through B, which is a conical flask containing a few c.cms. of cold water (all corks should be of rubber, and a rubber tube connection made at E). C is a similar flask containing a one-inch layer of lime-water. Any gas which escapes will pass on into D, and be collected over water. The flasks B and C should be in a trough of cold water, so that they may be cool during the operation. B serves to collect the tar and ammonia liquor, and C, containing lime-water, serves to "trap" CO_2, H_2S, and CS_2. In D, a fairly pure sample of coal gas will collect.

Comparison with a diagram of any coal gas plant will show that this is an experimental copy of the essential parts.

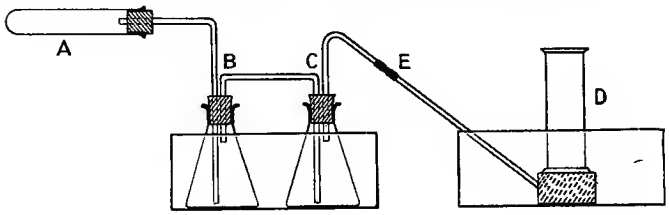

FIG. 44.

Before heating the test-tube A, make sure that all connections are gas-tight, and tap the tube lightly, so that a *clear channel* is left above the coal-dust for the escape of gases and vapours. Now gently heat the tube along its length with a smoky flame, and when hot gradually turn on the blue flame, and concentrate the heat on the front part of A, taking care not to burn the rubber cork. Smoke and vapours will soon be evolved, and bubbles of gas will pass through the two washing-bottles, B and C. Regulate the heat so that the gas bubbles are not too rapid, otherwise some of the tar and ammonia may be carried on and escape. When one jar is filled with gas, collect a fresh jar, and as the front portion of coal becomes exhausted, work the flame backwards. A *bright red-heat* should be used ultimately, but the glass must not be heated beyond the softening point, or it will probably "blow out," and the work will be spoiled. When all the coal has been exhausted, and no more gas is evolved, dis-

connect at E, and remove the cork from the heated tube A. Heating may then be stopped.

Determine the quantity of the products as follows:—

(*a*) From the volume of a gas-jar and the number collected, note down the volume in litres of coal gas generated.

(*b*) When the tube A, which contains the coke, is cool, weigh it, and so estimate the weight of coke produced.

(*c*) Take the flask B, which contains ammonia liquor and coal tar. Pour off the upper ammonia layer as completely as possible, into a beaker, and then pour into the flask a few c.cms. of water; shake round well, allow to settle, and pour off the top layer into the beaker. Repeat this once more. Place the beaker of ammonia liquor on one side, and pour the washed coal tar into a small weighed evaporating dish. Dry it well by dipping into it pieces of dry filter-paper, and when no more moisture is present, weigh the dish. This gives the weight of coal tar derived from the original weight of coal.

Knowing the weight of coke and coal tar in gms. given by a known amount of coal, calculate the weights of coal tar and coke, in pounds, which would be obtained from 1 ton of coal. Given that—

1 ton = 2240 lbs.
1 lb. = 454 gms. (approx.).

Also, calculate the gas evolved, in cubic feet per ton. Given that—

1 litre = 0·035 cubic foot (approx.).

Next, pour a few drops of the ammonia liquor into a test-tube, add a few drops of sodium hydroxide, and warm. A strong smell of ammonia is evolved, and a piece of red litmus placed in the tube is turned blue.

Now take the flask C, which contained lime-water to trap CO_2 and H_2S. Pour a few drops into a test-tube, and add a drop or two of hydrochloric acid. Effervescence shows a gas is evolved, and the smell is that of bad eggs. A glass rod moistened with lime-water and held in the gas is turned milky, and this proves that CO_2 is evolved.

The purification of coal gas by lime is dependent upon these reactions :—

(*a*) $$Ca(OH)_2 + H_2S = \underset{\text{Calcium sulphide.}}{CaS} + 2H_2O.$$

(*b*) $$Ca(OH)_2 + CO_2 = CaCO_3 + H_2O.$$

(*c*) $$CaS + CS_2 = \underset{\text{Calcium thio-carbonate.}}{CaCS_3}.$$

Finally, burn a jar of the collected gas, and note the luminous flame produced. Also compare the smell with that of ordinary coal gas.

The table on p. 182 shows in a clear manner how the coal tar is subsequently dealt with so that it may yield a large number of useful products, chief of which are benzene, carbolic acid, naphthalene, and anthracene.

The ammonia liquor is generally distilled with lime, and the liberated ammonia gas is absorbed in sulphuric acid to form ammonium sulphate, or it is passed into water and the strong solution sold as *liquor ammoniæ.*

Exp. 2. To Prepare Ammonia Gas.

Fit up a test-tube with a cork and obtuse-angled delivery-tube (Fig. 45). Place in the tube a few gms. of a mixture of powdered ammonium chloride and an equal quantity of powdered slaked lime. Fix the cork in position, and tilt the tube so that the delivery-tube, A, points vertically upwards. Warm gently, and show by a piece of moist red litmus paper that ammonia is given off. The smell confirms this, and if a glass rod moist with strong HCl be held near A, dense white fumes are formed, of ammonium chloride.

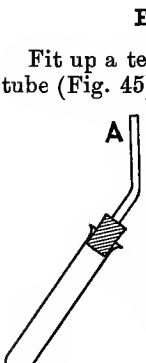

FIG. 45.

Equation—

$$NH_3 + HCl = NH_4Cl.$$

Now place an inverted test-tube over A, and collect a tubeful of the gas by upward displacement. Close the mouth of the tube with the thumb, and bring it under water in a dish. Then remove the thumb, and note how

rapidly the water fills the tube. This shows that ammonia is readily soluble in water. At 0° C., 1 volume of water dissolves over 1100 volumes of NH_3.

This explains why the gas cannot be collected over water, and the fact of collecting by upward displacement shows that the gas is less dense than air. Its density is roughly half that of air.

***Exp. 3. To Estimate the Percentage of Ash in Coal.**

Weigh between three and four gms. of finely powdered coal in a small porcelain dish. Place it on a pipeclay triangle, and put under it a small Bunsen flame. When the gases have burnt themselves out, increase the heat to redness and finally to bright redness. The carbon will be completely burnt away by this roasting, and the process should be facilitated after thirty minutes by clamping a draught-tube over the dish, as shown in Fig. 46. This tube has a length of about 20 cms. and a diameter somewhat less than that of the dish, and it should be clamped near to, or just inside the rim, to ensure a good draught of air for oxidising the carbon.

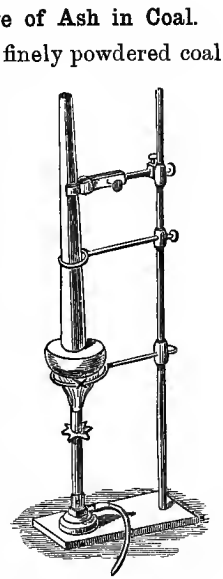

FIG. 46.—(*From Thorpe's "Quantitative Chemical Analysis."*)

The complete burning of the carbon may be further assisted by turning the ash over with a platinum wire towards the end of the process.

The time required for the completion of the oxidation is from one, to one and a half hours. When all the black particles have disappeared and the ash is white or tinged with red, remove the flame, allow the crucible to cool somewhat, and then transfer to a desiccator. When quite cool, weigh the ash.

***Exp. 4. To Estimate the Moisture in Coal.**

Well powder a small quantity of coal, quickly, and transfer between two and three gms., accurately weighed, to a

watch-glass, and clamp over this a second watch-glass, as in Fig. 22.

Place these, clamped together, in an air-oven, and heat to 110° C. for one hour. Then remove, place in a desiccator to cool, and weigh when cold.

Loss in weight = moisture.

Calculate from this result the percentage of moisture.

*Exp. 5. To Estimate Volatile Matter and Coke in Coal.

Weigh accurately about 2 gms. of powdered coal, in a crucible without the lid. Then place the crucible on a pipeclay triangle, cover with the lid, and heat it over a strong Bunsen flame. When the flame issuing under the lid disappears, continue heating for one minute, and then allow to cool. When somewhat cool, place the crucible in a desiccator to get cold, and then weigh without the lid.

Loss in weight = volatile matter + moisture.

Residue = coke = ash + fixed carbon.

When moisture, previously estimated, has been subtracted from total loss, the result = volatile matter.

Similarly, by subtracting weight of ash from total residue, the result = fixed carbon.

Note.—The destructive distillation of wood follows the same course as that of coal.

The products are: wood charcoal, wood tar, acetic acid, acetone, and wood spirit.

The gases evolved are non-luminous, and are utilised for heating the retorts.

Peat and shale when destructively distilled yield coke, tar, and ammonia liquor.

The gases in each case are burnt under the retorts.

Shale, in addition to these, yields a certain quantity of burning oils, known as shale oils.

The destructive distillation of bones yields bone charcoal, bone tar, bone oil, ammonia liquor, and gases which are burnt under the retorts.

Producer-Gas and Water-Gas.—Producer-gas is obtained by the action of air on red-hot anthracite. In Exp. 12, p. 74, the action of CO_2 on red-hot charcoal was proved

to yield the inflammable gas carbon monoxide. It is this gas which forms the combustible part of producer-gas, making up about 30 per cent. of the whole. The remainder is made up of about 60 per cent. nitrogen, and 10 per cent. CO_2 and hydrogen. Most of the CO_2, which may be formed at first, is quickly reduced to carbon monoxide by the red-hot coal in the producer, just as it was reduced in the experiment above mentioned.

Water-gas is formed by passing steam over red-hot anthracite, when the following reaction takes place—

$$C + H_2O = CO + H_2.$$

Nowadays this reaction is combined with the producer reaction, and the resulting gas, such as Mond gas or Dowson gas, has approximately the following composition—

$CO = 10{\cdot}2\ \%$. $CH_4 = 2{\cdot}5\ \%$. $H_2 = 26{\cdot}4\ \%$. $CO_2 = 16{\cdot}3\ \%$. $N_2 = 44{\cdot}6\ \%$.

The temperature of the producer is maintained at 1000° C., or slightly above. For the composition of producer gases, see p. 183.

*Exp. 6. To Prepare Water-Gas and Estimate its CO Content.

Fit up the apparatus used in Chap. II., Fig. 25 (passing steam over red-hot iron). In this case, however, fill the glass tube with granulated charcoal. Place the flask half-filled with water over a burner, and, while it is being heated to boiling, place a fan-burner under the charcoal tube, and gradually raise it to a bright red-heat. When a good jet of steam issues from the exit tube of the flask, connect on to the charcoal tube, and let the steam pass through the heated charcoal. Bubbles of gas will pass up through the water in the pneumatic trough, and two jars of the gas should be collected over water.

Prove, by a lighted taper, that the gas in these jars burns with a blue flame; and that CO_2 is formed during the combustion, by means of lime-water.

Next, fill a stoppered separating-funnel, cylindrical in shape (see Fig. 47), with water.

Invert it in the trough, stopper downwards, and then remove the stopper, and place the mouth over the beehive-shelf so that the issuing water-gas may fill the funnel. When quite full, replace the stopper, which should be greased, and keep the funnel of gas in a cool place for further use.

Now disconnect the apparatus at the junction of steam-flask and tube, and turn out the gas under both.

FIG. 47.

To estimate the volume of CO in the water-gas, hold the stoppered-funnel, previously filled, with the tube and tap uppermost, and pour into the tube cuprous chloride solution (see p. 164) till the tube is full.

Now carefully turn the tap, and allow some of the solution to enter the body of the funnel. Some of the carbon monoxide is absorbed, and more solution enters.

Great care must be taken that the solution does not rush in, taking air after it; therefore, when the cuprous chloride has nearly all passed in, close the tap securely.

Fill the tube up again, and let this also flow into the water-gas, and repeat till between 20 and 30 c.cms. of solution has been used.

Now close the tap, and, holding the stopper securely in position, turn the funnel so that the cuprous chloride becomes thoroughly mixed with the gas, and wets the side of the funnel. The funnel must be held by its ends, so that the heat of the hand does not alter the temperature of the gas, and after lying on its side for five minutes, it is again inverted and water poured into the tube.

The water is allowed to enter in the same way as the solution first used, and ultimately no more water will be able to pass in without gas passing out. This latter must be prevented, and when water just ceases to pass in, close the tap.

Now pour the liquid which entered the funnel into a graduated cylinder. The volume represents the volume of carbon monoxide absorbed by cuprous chloride.

By filling the funnel completely with water, its total

volume may be determined, and thus the percentage of CO present, estimated.

The remainder of the gas will be mainly hydrogen, with a small amount of CO_2.

Above 1000° C. the reaction takes place almost entirely according to the equation—

$$C + H_2O = CO + H_2.$$

Below 1000° C. the amount of CO_2 increases as the temperature falls, and at 600° C. the following reaction only takes place—

$$C + 2H_2O = CO_2 + 2H_2.$$

The charcoal tube should therefore be maintained at bright redness during the experiment.

*Exp. 7. Fractional Distillation of Petroleum.

Crude petroleum is fractionally distilled, on an industrial scale, from iron retorts. As the temperature rises, the various fractions which distil over are collected in separate receivers, and in this way the following useful products are separated from the crude material :—

50° C. to 70° C.	Petroleum ether.	Light oils used as solvents.
70° C. to 90° C.	Benzine.	
90° C. to 120° C.	Ligroin.	
120° C. to 150° C.	Cleaning oil.	
150° C. to 300° C.	Illuminating oils (kerosene).	
Above 300° C.	Lubricating oils.	

Residue = carbon.

Experimental.—Using the apparatus figured below (Fig. 48), submit the specimen of petroleum supplied, to fractional distillation, collecting the distillate in three fractions—

First fraction : Light oils up to 150° C.

Second fraction : Illuminants 150° to 300° C.

Third fraction : Lubricants above 300° C.

Fill a small distilling-flask A, not more than two-thirds full with 100 gms. of the petroleum. Place a piece of porous pot in the flask to prevent " bumping," and fix a thermometer reading to 400° C. in the neck of the flask by a single-bored

cork, so that the thermometer bulb is just below the side opening of the flask. Join the flask on to a water-condenser, which is clamped on a retort-stand, and through which water passes by the rubber connection shown in the figure.

Three small beakers or flasks, of known weight, are required for collecting the distillate.

Heat the flask on a sand-bath, and collect in the first beaker, the fraction which distils while the thermometer in the flask is rising to 150°. Light oils.

When the thermometer registers 150° C., change the receiver and proceed to collect all that distils between 150° and 300° C. Illuminating oils.

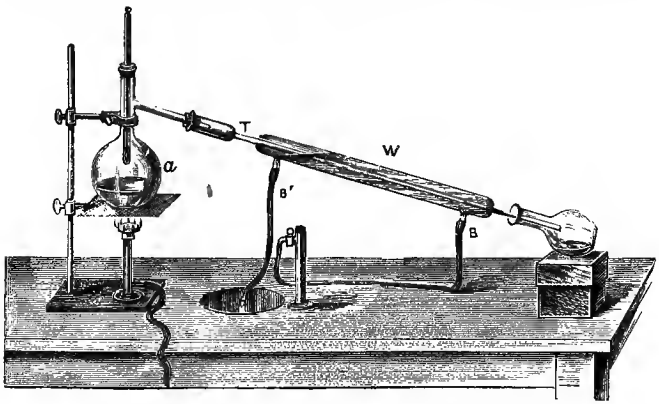

FIG. 48.—(*From Newth's "Elementary Practical Chemistry."*)

When the thermometer reaches 150° the current of cooling water running through the condenser should be stopped and the water run out.

After reaching 300°, change the receiver again, and collect the portion which distils above 300°, *i.e.* the lubricating oils.

When nothing more distils over, cease heating the flask, which in these last stages (above 300°) may be heated *directly* with the flame.

Weigh the three fractions collected, and register the percentage of each yield in your note-book.

The difference between the sum of these three and the

original 100 gms. represents the percentage yield of carbon which forms a residue in the distilling-flask.

Exp. 7a. Determination of Flash-Point by the "Open Test."

Place about 50 c.cms. of the oil in a small wide-mouthed flask or small dish, so as to fill it about three-fourths full.

Embed the vessel in a sand-bath to slightly above the liquid level, and insert a thermometer in the oil. Heat the sand-bath gently so that the temperature rises gradually, and bring a small flame, from time to time, to the surface of the oil.

Note the lowest temperature at which a "flash" takes place. This is the flash-point, and will be correct to within between 2° and 5° C.

A second determination should be made, and the temperature of the bath raised very slowly in the neighbourhood of the first reading.

Note.—The flash-point may be taken with greater accuracy by what is known as the "close test." For this, one of the standard forms of flash-point apparatus must be used. The student is referred to larger text-books dealing with this subject.

Lubricating Oils.—In an examination of lubricating oil the following tests are usual :—

1. *Determination of "Gravity."*—This determination may be carried out with the aid of a specific gravity bottle, and the "gravity" should be determined to the fourth decimal place.

The specific gravity is a most important physical constant of a specimen of oil, and for that reason is always determined.

2. *Estimation of Animal, Vegetable, and Mineral Oil.*—It is important to know the amount (if any) of animal or vegetable oil present in a lubricant. The presence of this kind of oil leads to the formation of organic acids, and ultimately to undue corrosion of the machine parts on which the lubricant is used.

Hydrocarbon or mineral oils are to be preferred, because they do not yield organic acids, and hence produce the minimum amount of corrosion.

For an account of "cylinder deposits" caused by lubricating oil, see Table XXII.

For the estimation of animal or vegetable oil in lubricant, see Exp. 11.

3. *Cold Test.*—Seeing that a lubricant may be used in cold weather for outdoor work, it is important that it should not solidify or lose its fluid state under working conditions.

The cold test involves a determination of the temperature at which the oil just begins to flow easily, and is carried out as follows:—

***Exp. 8.**—Place 50 c.cms. of the oil in a small bottle without a stopper, and immerse the whole in a freezing mixture (see Table VII.). Allow the oil to solidify, and then remove the bottle from the freezing-bath.

Let the temperature rise, and keep a thermometer in the oil so that the temperature can be read. Turn the bottle about, and keep the oil stirred with the thermometer when the oil shows signs of melting. Note carefully, the exact temperature at which the oil begins to flow easily.

A good mineral lubricant will not change its consistency appreciably between 10° and 25° C.

If, however, any animal or vegetable oils are present, a marked change in the "flow" will be manifest when the temperature falls to 10° C.

4. *Viscosity.*—This property of a lubricant, which has reference to the ease with which the liquid particles or layers slide over or past one another, is obviously of great importance, seeing that the lubricant is used to diminish friction.

The viscosity is measured relatively to that of water by the following method:—

***Exp. 9.**—Fifty c.cms. of water, at the temperature of the room, are allowed to run from a vertical tube, through a narrow orifice at the lower end, having a diameter of 1 to 1·5 millimetres. (A burette is suitable.)

The time taken, in seconds, for this amount of water to run out is measured.

Next, the oil under question is run out in exactly the same way, and the time, in seconds, measured which is required for 50 c.cms. to escape.

The ratio of the two times, taking that for water as unity, gives the relative viscosity of the oil.

Note.—The temperature has a marked effect upon viscosity, and for this reason very exact determinations of this value must be carried out

in some standard apparatus, such as Engler's or Redwood's. (See text-books.)

5. *Flash and Fire Tests.*—It is important to know the temperature at which a lubricant will flash, and also the higher temperature at which it will burn.

Exp. 10.—Flash and fire test is carried out in the same manner and with the same apparatus as in Exp. 7a. When the flash-point has been taken, allow the temperature of the sand-bath to rise slowly, and note the reading of the thermometer when the oil first continues to burn after the flashing-flame has been removed.

6. *Acidity of Lubricating Oil.*—The acidity can be detected, if present, by dissolving a few drops of the oil in acid-free alcohol, and adding two or three drops of phenol-phthalein dissolved in alcohol.

If the oil is neutral, on adding two drops of decinormal potash solution the indicator will become pink.

If, however, "acidity" is present, a certain quantity of the potash solution will be required before the mixture becomes neutral, and one more drop gives a pink colour.

The quantitative estimation of acidity is carried out, as above, by using a measured volume of the oil in acid-free alcohol, and adding standard alcoholic potash until the pink colour is obtained.

The acidity may be expressed in terms of oleic acid, taking as a basis that 1 c.cm. of normal potassium hydroxide is equivalent to 0·282 gm. of the acid.

Exp. 11. Estimation of Percentage of Mineral Oil in Lubricant.

Mix 10 gms. of the oil with 75 c.cms. of alcoholic potash (60 gms. KOH in 95 per cent. alcohol, made up to 1 litre), and evaporate the mixture in a dish, on the water-bath, until all the alcohol is expelled.

At the end of this time, any animal or vegetable oils will have been saponified by the potash and converted into soap.

Next, add 75 c.cms. of water, stir well to ensure the complete solution of the soap, and then transfer to a separating funnel. Add 75 c.cms. of ether, cork up, agitate well, and

then stand the funnel in an upright position for half an hour (for greater accuracy it should stand 12 hours).

At the end of this time two layers are present in the funnel, the upper of which contains the mineral oil dissolved in ether. Run the lower layer off as completely as possible, then pour the ethereal solution into a weighed flask, evaporate the ether on a water-bath, and weigh the residual mineral oil.

Exp. 12. Turpentine Oil.

This oil is obtained from the resinous juices which exude from many coniferous trees, most notably the pines.

The resinous exudations are distilled with steam, when the turpentine oil, which is volatile, separates from the resins which remain behind. Its chief use is for the preparation of varnishes and oil-colours.

The following determinations are usually carried out to decide upon the quality and purity of the turpentine :—

1. *Specific Gravity.*—This value should lie between 0·862 and 0·875.

2. *Distillation.*—The boiling-point should be about 156° C., and 95 per cent. of the oil should distil over between 153°·5 C. and 165°·5 C.

This test should be carried out in a small distilling-flask using 100 c.cms. of turpentine.

3. *Residue on Evaporation.*—Evaporate 10 gms. in a dish on a water-bath. The residue should amount to less than 2 per cent.

4. *Test for Mineral Oils.*—Place 6 c.cms. of the turpentine in a 50-c.cm. thin-walled tube with stopper, graduated in c.cms. and tenths. Stand the tube containing the oil, in cold water, and add slowly to it, a mixture containing 4 parts of concentrated H_2SO_4 and 1 part of fuming H_2SO_4.

Shake up after each addition, keep cool, and finally, after adding 20 c.cms. of the acid mixture, let the tube cool, and stand for half an hour. The layer of oil which collects on the top represents mineral oil, and should not amount to more than 0·3 c.cm. if the turpentine is pure.

CHAPTER IX

SULPHITES AND SULPHATES—NITRITES AND NITRATES—CHLORATES

*Exp. 1. To Burn Sulphur in Air and Study the Gas Formed.

Take two gas-jars, fitted with glass plates, and place them on the bench. Then fill a deflagrating spoon with powdered sulphur. Ignite the sulphur by heating the spoon in a Bunsen flame, and when burning, place the spoon in the first jar, and allow it to burn out.

When it has burnt out, remove it, put on the glass cover of the jar, and relighting the sulphur that still remains in the spoon, place it in the second jar, and let it burn out once more. These two jars will now contain sulphur dioxide. Dip into the first jar a piece of paper, moistened with potassium chromate, and note that it quickly becomes green. Note also the pungent odour of the gas. Bring to the mouth of the jar a lighted taper or match; the gas does not burn, and on pushing the light into the gas it is extinguished. This gas, therefore, does not burn, neither does it support combustion. Four-fifths of the jar contains nitrogen, which is not attacked by the burning sulphur.

Now take the second jar, invert it under water, and remove the glass cover. Note from the rise of level in the jar that the gas is soluble in water, and while still under, replace the cover, and then take the jar out. Place it upright on the bench, and pour into it a little neutral litmus solution. The colour is changed to red. This shows that the solution of sulphur dioxide in water is acid. Such a solution is known as sulphurous acid. At 0° C., 1 volume of water is capable of dissolving nearly 8 volumes of SO_2.

The gas is made commercially either by the combustion of sulphur in air, or by the combustion of iron pyrites.

$$\underset{\text{Iron pyrites.}}{2FeS_2} + 11O = \underset{\text{Ferric oxide.}}{Fe_2O_3} + \underset{\text{Sulphur dioxide.}}{4SO_2}.$$

Sulphuric acid (oil of vitriol) is manufactured by allowing the sulphur dioxide gas thus formed to react with oxygen and water, when the following change takes place :—

$$SO_2 + O + H_2O = \underset{\text{Sulphuric acid.}}{H_2SO_4}.$$

Two processes are used—the older "chamber process," in which SO_2, oxygen and steam react in lead chambers; and the more modern process, where sulphur dioxide and oxygen combine in the presence of finely divided platinum, *i.e.* "the contact process." The resulting sulphur trioxide is then passed into water, with which it forms sulphuric acid.

(*a*) $$SO_2 + O = SO_3.$$

(*b*) $$SO_3 + H_2O = H_2SO_4.$$

Exp. 2. To Prepare Sulphur Trioxide and Sulphuric Acid.

This is to be accomplished by passing air over heated iron pyrites, and then the resultant sulphur dioxide is to be passed with air over heated platinised asbestos (see p. 163). The resultant SO_3 will be condensed in water.

The apparatus to be used is shown in Fig. 49.

A is a Winchester-quart which serves as an air-holder. By running water from a funnel connected at *b*, by rubber, air is driven through the right-angled tube and through B and C. These two sections of the tube are both 10 to 18 cms. long, they are joined by a drawn-out piece of smaller diameter, and each open end is closed by means of a cork. A right-angled tube leads from C to a Woulffe's bottle, and this is connected to a similar bottle, so that the gases traverse first one bottle and then the other.

Both Woulffe's bottles stand in a trough containing cold water, which acts as a cooling-bath.

A small quantity of distilled water is placed in each bottle to about the depth shown in the figure. The tubes entering these bottles should have the same lengths as in the figure.

When the apparatus is fitted together and made gas-tight, weigh accurately about 10 gms. of iron pyrites, and pack it, in small pieces, into the tube B. The portion C must be loosely filled with platinised asbestos. When the tubes are filled and each part of the apparatus is in position, gently heat the platinum in C, and, while a *slow stream of air* is

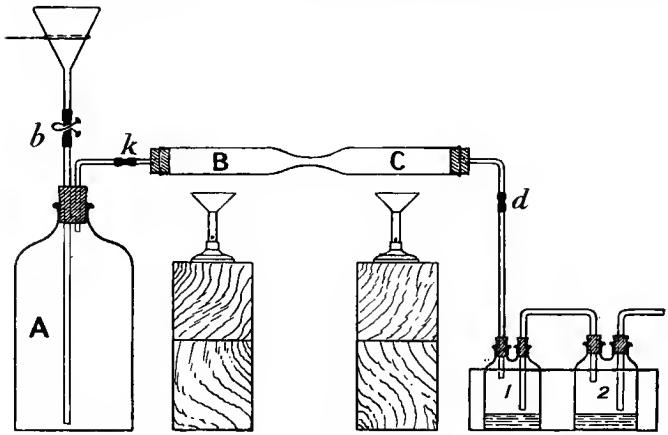

FIG. 49.

passing, gradually raise the temperature to a dull red-heat. Just before this temperature is reached in C, commence to heat the pyrites in B, and contrive that it reaches a dull red-heat shortly after C. Then pass air more rapidly, and when one "Winchester" is used up, disconnect at *k* and put on a fresh one. The gases now passing through the first Woulffe's bottle will consist of SO_2, and air which is unused (mainly nitrogen). Under proper working conditions, a high percentage of the SO_2 should be oxidised to SO_3, in the tube C, and this, on volatilising into the Woulffe's bottle, will dissolve in the water.

After thirty minutes, when the air from six "Winchesters"

has been used, all the iron pyrites will be burnt, and the sulphur trioxide completely driven over into the water. Now disconnect at *k*, and remove the burners. Bottle 1, will contain all the sulphuric acid, unless the gas has passed too rapidly. To ascertain if there is any sulphuric acid in 2, pour a few c.cms. into a test-tube, add two or three drops of hydrochloric acid, and then a few drops of barium chloride solution. A white ppt. indicates sulphuric acid, and in this case mix the liquid in both bottles in a beaker. If, however, no H_2SO_4 is found in the second bottle, reject its contents as of no value.

Dip a piece of paper, moist with K_2CrO_4 (potassium chromate), into the liquid. If the paper turns green, the liquid contains SO_2, and must be boiled until chromate paper is not changed. Prove that sulphuric acid is present in the boiled liquid by removing a drop on a glass rod to a watch-glass, and adding to it one drop of barium chloride.

Transfer the hot liquid to a 250-c.cm. flask, wash the beaker once with distilled water, and add the washings to the flask.

Cool this by holding under a cold-water tap, and when cold, dilute to the mark. The sulphuric acid obtained from the weight of pyrites used may now be estimated by withdrawing 10 c.cms. and titrating it with standard alkali.

From this result calculate the quantity of sulphuric acid obtainable from 1 kilogram of pyrites (see Chapter XII.).

Exp. 3. Further Methods for Preparing SO_2.

(*a*) By the reducing action of metallic copper upon concentrated H_2SO_4.

Place a few copper turnings in a test-tube, and cover them with concentrated H_2SO_4. Warm the contents of the tube, and finally, heat strongly. Observe the sulphurous odour of the gas evolved, and prove it is SO_2, by dipping into the mouth of the tube, a piece of paper stained yellow with potassium chromate. A green colour shows the presence of SO_2.

Equation—

$$Cu + 2H_2SO_4 = CuSO_4 + SO_2 + 2H_2O.$$

(*b*) Place in a test-tube a little powdered sulphite of sodium or calcium. Pour on to this, dilute hydrochloric acid, and shake. Effervescence takes place, and a gas is evolved having the odour of SO_2. Place a piece of "chromate paper" in the gas to confirm the presence of SO_2.

Equation—

$$Na_2SO_3 + 2HCl = 2NaCl + H_2O + SO_2.$$

***Exp. 4. To Prepare Nitric Acid from Saltpetre.**

Saltpetre occurs in large quantity in Peru and Chile. It is largely used as a nitrogenous manure, and for preparing nitric acid.

Fit up an 8-oz. retort, as was done, for distilling water in Exp. 4, p. 67. Weigh out accurately about 40 gms. of saltpetre (sodium nitrate). Place it in the retort, and add about 30 gms. (16 c.cms.) concentrated H_2SO_4. Shake round well, replace the stopper, and fix in position with a small weighed flask for receiver. This receiver must be kept cool by immersion in cold water. Now heat the retort with a small flame. The reaction soon commences, and the upper part of the retort and neck become filled with brown vapours. These partially condense and collect in the receiving flask as a pale yellow liquid. Continue heating till no more nitric acid distils over, increasing the heat if necessary. When the decomposition is complete, remove the flame, and also remove and cork the receiver. While still molten, the "nitre cake" remaining in the retort should be poured out of the tubule into a weighed evaporating basin, and when cold, its weight ascertained. Calculate from this experiment the weight of nitre cake and nitric acid which could be obtained from 1 cwt. of saltpetre, using sulphuric acid in the same proportion as above.

***Exp. 5. Tests for Nitric Acid and Nitrates.**

(*a*) Pour a few c.cms. of the nitric acid prepared as above, into a test-tube, and drop in a few copper turnings. Vigorous reaction ensues, and a brown-red gas is evolved. The solution in the tube rapidly assumes a blue colour owing to the formation of copper nitrate.

(*b*) Into a second tube containing another sample of the nitric acid, drop a piece of tin. Vigorous action follows, and red-brown fumes are evolved. The solution in this case deposits white hydrated tin dioxide.

(*c*) *Test for Nitrate.*—To a little powdered potassium or sodium nitrate contained in a test-tube, add a few drops of strong sulphuric acid, warm the mixture gently, and then drop in a copper turning. Reddish-brown fumes are at once evolved, and the solution turns blue. In this test the copper is attacked by the free nitric acid, which is liberated by the interaction of concentrated H_2SO_4 and a nitrate.

Exp. 6. Action of Heat on Nitrates.

(*a*) Place a few crystals of potassium nitrate in a hard glass tube, and heat strongly. The crystals melt, and ultimately, bubbles of gas are evolved from the molten nitrate. Into the gas push a glowing splinter, and observe its rekindling. The gas evolved by heating potassium nitrate is oxygen, and the residue left in the tube is potassium nitrite.

Equation—

$$2KNO_3 = 2KNO_2 + O_2.$$

Add to a little potassium or sodium nitrite in a test-tube, a few drops of dilute hydrochloric acid. Brown fumes are immediately evolved with effervescence, even in the cold. This reaction is a test for nitrites, and distinguishes these salts from the nitrates.

(*b*) Heat a few powdered crystals of lead nitrate in a hard glass tube. There is considerable crackling, and brown fumes of nitrogen peroxide are given off. That oxygen gas is evolved as well may be proved by pushing a glowing splinter into the gas. A rekindling shows the presence of oxygen. The residue is, on cooling, a yellow colour, and is lead oxide.

Equation—

$$\underset{\text{Lead nitrate.}}{2Pb(NO_3)_2} = \underset{\text{Lead oxide.}}{2PbO} + \underset{\text{Nitrogen peroxide.}}{4NO_2} + O_2.$$

(*c*) Heat a few crystals of copper nitrate in the same manner. The salt is completely decomposed, brown fumes

are evolved, and copper oxide (a black powder) remains behind.

Equation—

$$2Cu(NO_3)_2 = 2CuO + 4NO_2 + O_2.$$

The nitrates and carbonates of the common metals are all decomposed at a red heat, and the oxide of the metal is left behind.

Exp. 7. Nitrates and Chlorates.

Heat a very little potassium nitrate on charcoal in a blow-pipe flame. The charcoal deflagrates owing to the oxygen which is liberated by heat.

Repeat this experiment with a little powdered potassium chlorate (chlorate of potash). The same effect is produced, and for the same reason. When potassium chlorate is heated it melts at about 370° C., and at a higher temperature it decomposes into oxygen and potassium chloride.

Equation—

$$\underset{\text{chlorate.}}{\underset{\text{Potassium}}{2KClO_3}} = \underset{\text{chloride.}}{\underset{\text{Potassium}}{2KCl}} + 3O_2.$$

*Exp. 8. To Prepare Oxygen by Heating Chlorate of Potash.

Fit up a hard glass test-tube with single-bored cork and delivery-tube. Arrange it by means of a retort-stand and clamp so that the evolved gas may be collected over water in a trough (see Fig. 28). Weigh the dry tube, and place in it 1 gm. exactly, of powdered and dry potassium chlorate. Fit the cork and delivery-tube in position, and by heating, expel the oxygen gas, and collect it in a jar over water. When, by strong heating, no more gas is evolved, remove the cork from the tube, and allow it to cool. While the residue is cooling, test the jar of collected gas by means of a glowing splinter, to prove it is oxygen.

When the tube is cold, weigh it, and from the loss in weight, calculate the weight of oxygen evolved from 1

kilogram of potassium chlorate. Compare this result with the calculated value derived from the above equation.

Note.—Chlorate of potash is prepared by passing chlorine gas through a solution of potash in water, or by the electrolysis of an aqueous solution of potassium chloride.

Equation—

$$3Cl_2 + 6KOH = 5KCl + KClO_3 + 3H_2O.$$

Exp. 9. To Estimate the Constituents of Gunpowder.

Gunpowder consists of nitre (potassium nitrate), 75 per cent., mixed with nearly equal quantities of powdered charcoal and sulphur.

Nitre is soluble in water. Carbon and sulphur are not.

Sulphur is soluble in carbon bisulphide. Nitre and carbon are not.

By making use of these two solvents, carbon bisulphide and water, the three ingredients of gunpowder can be separated, and the weight of each estimated.

Weigh accurately about 3 gms. of the powder in a fine state of division, place it in a small porcelain dish, and fit a funnel and weighed filter-paper in a stand, ready for filtering. Take about 20 c.cms. of carbon bisulphide (*don't bring it near a flame*), and pouring half of it on to the powder, triturate it well with a glass rod. After settling, pour the liquid through the weighed filter-paper. When the liquid has run through, pour on the rest of the carbon bisulphide, and after triturating, pour the whole (solid and liquid) on to the filter-paper.

Now pour the clear filtrate on to the filter-paper and its contents, and let it run through. Repeat this operation twice, so that all the sulphur may be completely extracted from the powder. The residue consists of nitre and charcoal.

Place the filter and funnel in the steam-oven for ten minutes.

By that time all the carbon bisulphide will have vaporised, and the contents will be quite dry. Remove the filter-paper, and weigh it. The increase on its original weight gives the weight of charcoal and nitre present. By subtracting this weight from that of the original powder the amount of sulphur is obtained.

If the carbon bisulphide has not been thrown away, it should be at once thrown down the sink in a fume-cupboard. Take the filter containing the charcoal and nitre, and tip the contents carefully into a small porcelain dish. Replace the filter-paper in its funnel in the filter-stand.

Add to the contents of the dish small quantities of warm water (20 c.cms. at a time), and stir well with a glass rod. Use three separate portions of water, and after each stirring and settling, decant the clear liquid on to the filter-paper. Now add a fourth quantity of warm water, stir well, and, without allowing to settle, pour the whole on to the filter-paper. Any charcoal still remaining in the dish should be now removed to the filter by a stream of warm water from a wash-bottle. When all the water has run through, remove the funnel and filter (now containing charcoal only) to the steam-oven, and leave till quite dry. When dry, weigh it, and from the further loss in weight ascertain the amount of nitre and charcoal present in the sample of powder.

Calculate from these experimental results the percentage composition of the sample of powder.

Table showing Composition of various Powders.

	Nitre.	Charcoal.	Sulphur.	Moisture.	Potassium sulphate.
Black rifle powder .	75	15	10	...	...
Spanish powder .	75·30	11·34	12·42	0·65	0·29
Large grain rifle .	74·95	13·52	10·27	1·11	0·15

*Exp. 10. To Prepare Nitrogen by Heating Ammonium Nitrite.

Ammonium nitrite is conveniently prepared in solution, by the action of sodium nitrite upon ammonium chloride. The following reaction takes place:—

$$NaNO_2 + NH_4Cl = NaCl + NH_4NO_2.$$

The ammonium nitrite is in solution together with sodium chloride, and when such a solution is heated gently, the former salt decomposes into nitrogen and water.

Equation—

$$NH_4NO_2 = 2H_2O + N_2.$$

Fit up a 4-oz. conical flask with thistle-funnel and delivery-tube as shown in Fig. 50. Place in the flask, about 5 gms. of sodium nitrite and 5 gms. of ammonium chloride. Replace the cork, and add through the thistle-funnel about 50 c.cms. of water. Shake round, and when the salts have dissolved, giving a clear solution, warm gently. Bubbles of gas at once make their way through the water in the pneumatic trough, and when the air in the apparatus has been displaced, proceed to collect two jars of nitrogen. When the jars have been filled, disconnect the delivery-tube, and stop warming the flask.

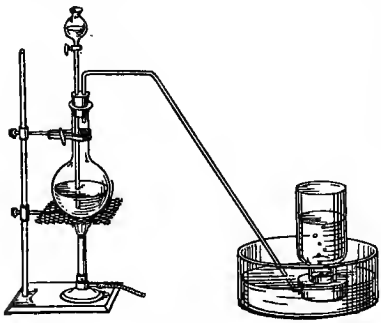

FIG. 50.—(*From O'Shea's "Chemistry for Coal-Mining Students."*)

Prove that the collected gas is nitrogen as follows:—

Jar 1.—Observe that the gas is colourless and odourless. Plunge into the jar a lighted taper, and note that it is immediately extinguished. The gas itself does not burn, neither does it support combustion.

Jar 2.—Pour into this jar a little clear lime-water, and shake well, after covering the jar with a glass plate. Observe that no turbidity is produced, a characteristic which distinguishes nitrogen gas from carbon dioxide.

CHAPTER X

COMPOUNDS OF NON-METALS: PHOSPHORUS, ARSENIC, SILICON — COMPOUNDS OF METALS: CHROMIUM, TUNGSTEN, MANGANESE—INORGANIC SUBSTANCES USED AS PIGMENTS

Exp. 1. Phosphorus. Phosphoric Acid. Phosphates.

PLACE a gas-jar (clean and washed out with distilled water) on the bench. Fix a *small* piece of phosphorus, the size of a pea, on a deflagrating spoon, light it, by holding it for a second in the Bunsen burner, and quickly transfer the spoon with the burning phosphorus to the gas-jar, and allow it to burn out. White fumes are formed, which condense on the inside of the jar, and which consist mainly of phosphorus pentoxide (P_2O_5).

Now remove the spoon, and cool it by dipping it into cold water. Pour into the gas-jar, about 20 c.cms. of cold distilled water, and shake up well, so as to dissolve all the white deposit. Filter the liquid (if necessary), and pour a few drops of the clear solution into a test-tube containing neutral litmus solution.

The litmus assumes a red tint, indicating that the phosphorus oxide solution is acid. It contains actually, phosphoric acid formed by the solution of phosphorus pentoxide in water.

Equation—

$$P_2O_5 + 3H_2O = 2H_3PO_4.$$

Next, pour a few c.cms. of the clear acid solution into a test-tube, and then add to it a few drops of *magnesia mixture* (see p. 163), and shake well. A white crystalline ppt. is formed.

Repeat this test, using sodium phosphate solution, in

another test-tube. A similar white crystalline ppt. is produced. This is an important test for phosphoric acid and phosphates.

*Exp. 2. To Prepare Calcium Phosphate by Burning Bone.

Wrap a small piece of bone in a platinum wire by rolling the wire round so that the bone can be held in a Bunsen flame. Now hold the bone in the flame, and heat it till it begins to burn. Inflammable gases and vapours are given off, and when these cease, a black mass of bone charcoal is left. Continue heating, and the charcoal will glow and gradually burn away, leaving a white ash, which can be easily powdered. Transfer the residual ash (calcium phosphate) to a small evaporating dish, add 20 c.cms. of distilled water and 5 to 10 drops of dilute hydrochloric acid, and stir with a glass rod. The phosphate will completely dissolve. Pour a few c.cms. into a test-tube, and add *magnesia mixture* with shaking. A white crystalline ppt. proves the presence of *phosphate*.

Dip a platinum wire into the remainder of the solution, and then bring it into the Bunsen flame. A fine brick-red flame shows the presence of *calcium*.

*Exp. 3. Arsenic.

The most common compound in which arsenic occurs is known commercially as "white arsenic." It is the oxide of the element, and has the composition As_4O_6. It is formed in the roasting process of many metallurgical operations, when ores containing arsenic are dealt with. The chief uses of white arsenic are for glass-making and calico-printing.

(*a*) Heat a little piece of arsenic in a small ignition-tube. Note how it sublimes, and collects in a metallic-looking deposit on the cool parts of the tube.

(*b*) Heat a little arsenic on charcoal (in a scoop) by means of a blowpipe flame. Small clouds of white arsenic (arsenic oxide) are formed, which partly volatilise and partly form a white incrustation on the charcoal. A garlic odour accompanies these fumes, and is one of the tests for arsenic.

(*c*) Dissolve a little arsenic oxide in dilute hydrochloric

acid. Transfer the solution to a boiling-tube, and put in two or three strips of clean metallic copper. Boil for two minutes, and at the end of this time the copper will be covered with a grey film of arsenic. If the strips be now removed and dried between filter-paper, and put in a clean dry test-tube; on heating, the arsenic becomes sublimed and oxidised, so that a white deposit of As_4O_6 collects on the cooler part of the test-tube.

(*d*) Take another portion of hydrochloric acid solution of white arsenic, and bubble hydrogen sulphide gas through it. A yellow ppt. is produced, of arsenic sulphide.

Equation—

$$2AsCl_3 + 3H_2S = As_2S_3 + 6HCl.$$

Exp. 4. Marsh's Test for Arsenic.

This is a very delicate test for arsenic, and is carried out as follows :—

Fit up a 4-oz. conical flask with thistle-funnel and right-angled tube, as shown in Fig. 51. The tube should be of hard glass, and should be drawn out at the end. Place in this flask a few pieces of zinc (arsenic free), and just cover them with distilled water. Then replace the cork, and pour a few drops of strong hydrochloric acid through the thistle-funnel. Hydrogen gas will be evolved, and while the air is being expelled from the apparatus, dissolve a little arsenic oxide in dilute hydrochloric acid.

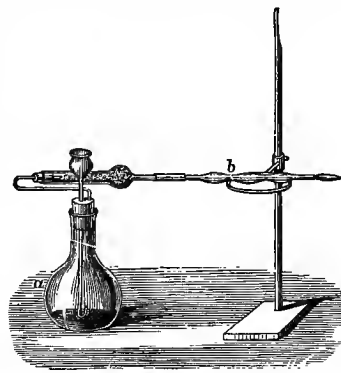

FIG. 51.—(*From Thorpe's "Qualitative Chemical Analysis."*)

Now ascertain whether the hydrogen is air-free by collecting a test-tube of the gas by upward displacement, closing the mouth of the tube with the thumb, and bringing it mouth downwards to a flame. If the gas burns quietly it may be lighted at the end of the

tube, and continues burning with a blue flame. Now pour a few drops of the arsenic solution through the thistle-funnel, and observe the following :—

(1) The flame burning at the end assumes a livid-blue tint, and if a cold dish be held against it, a black deposit of arsenic is obtained.

(2) Heat the hard glass tube gently at *b*, and observe that just beyond the heated portion a black deposit of arsenic is obtained.

***Exp. 5. To Test for the Presence of Silica.**

Place a little of the powdered silicate in a small lead or platinum dish, mixed with an equal quantity of powdered fluor-spar (calcium fluoride).

Add a few drops of concentrated H_2SO_4 and heat gently.

If now a metal or wooden rod, with a drop of water on the end, be held in the fumes, the clear drop will become turbid, because of the silica which is deposited therein.

Try the above test with small quantities of powdered glass and cement.

Silica and Silicates.—Silica (silicon oxide) or rock-crystal forms, together with the silicates, the major portion of the earth's crust. In the form of sand and glass it is a common material, and the important minerals in which it is a constituent are given in table, p. 175.

One of the most important industrial applications of silica is in the preparation of mortars and cements in which it occurs, combined with the bases lime, potash, and soda.

A scheme for the testing and analysis of cements and mortars will be found on p. 156.

Chromium and Chromium Compounds. — Chromium, a metal resembling iron in properties, but with a much higher melting point, is important as a constituent of certain steels. The metal itself is prepared from the oxide Cr_2O_3, either by roasting with carbon in the electric furnace or by reduction with aluminium powder (thermite reaction), see p. 106.

Exp. 6. To Prepare Potassium Chromate.

Mix in an iron crucible the following substances, well dried and powdered: 15 gms. chromium oxide, 7 gms. potassium carbonate, 30 gms. potassium nitrate.

This mixture should fill not more than two-thirds of the crucible in order to avoid frothing over.

Cover the crucible and heat it, either over the blowpipe or in a furnace. The heating must be continued until gases cease to be evolved, and the mass is in a quietly molten condition. The following change has taken place :—

$$2Cr_2O_3 + K_2CO_3 + 6KNO_3 = 4K_2CrO_4 + CO_2 + 3N_2O_3.$$

Remove the crucible by tongs, and stand it on a retort-stand base, or a piece of sheet iron. This will facilitate cooling, and cause the "melt" to draw away from the sides of the crucible. As soon as it is somewhat cool, drop the crucible into a dish of distilled water, boil the water, and agitate the crucible contents, so that the "melt" is completely transferred to the water.

The yellow solution contains potassium chromate. Filter from any dirt or insoluble residue into an evaporating dish, and evaporate to crystallisation. Drain the crystals, dry between filter-paper, and weigh them.

The calculated yield is 40 gms., and the actual yield should be between 30 and 40 gms.

Exp. 7. Chromium Hydroxide.

Dissolve a few crystals of potassium chromate in water, and add to the solution 2 or 3 c.cms. of sulphurous acid.

The yellow colour changes to green, owing to the reduction of the chromate to chromium sulphate.

Equation—

$$2K_2CrO_4 + 5H_2SO_3 = Cr_2(SO_4)_3 + 2K_2SO_3 + 5H_2O.$$

Now add a little ammonium hydroxide to the solution, and a blue-green ppt. of chromium hydroxide will be formed.

Equation—

$$Cr_2(SO_4)_3 + 6NH_4OH = 2Cr(OH)_3 + 3(NH_4)_2SO_4.$$

*Exp. 8. Tungsten and Tungsten Oxide.

The mineral from which tungsten is generally obtained is *wolfram*, a tungstate of iron and manganese having the composition $(MnFe)WO_4$. From this, tungstic oxide (WO_3) can be prepared by roasting with fusion mixture.

Mix a small quantity of powdered *wolfram* with five or six times its weight of fusion mixture ($Na_2CO_3 + K_2CO_3$) in a porcelain crucible. Cover with the lid, and heat over the blowpipe, or in a muffle furnace. When the melt is quiet and all bubbling has ceased, remove the crucible to an iron plate, and allow to cool. While still hot, place the crucible in a dish of distilled water and extract the melt thoroughly. Filter the hot solution from undissolved iron and manganese oxides.

The solution contains *sodium tungstate* (Na_2WO_4).

Divide this into two parts. To one part add a few drops of concentrated HCl. A white ppt. of *tungstic acid* is produced.

To the other part add a piece of zinc and a few drops of dilute HCl. Hydrogen is evolved, and the solution becomes blue in colour owing to the reduction of the tungstic acid.

From tungstic oxide the metal can be prepared by heating with aluminium powder (thermite reaction).

It is of great value in preparing *tungsten steel* for tool-making.

*Exp. 9. To Prepare Manganese from its Oxide by the Thermite Method.

The chief ore of manganese is *pyrolusite*, which consists mainly of the oxide MnO_2.

Take 50 gms. of powdered pyrolusite, and ignite it to a red-heat in a crucible, so that it becomes converted into Mn_3O_4. This contains less oxygen than the pyrolusite, and hence the reaction with aluminium is not so violent.

The amount of Mn_3O_4 given is approximately 42 gms., and this must be mixed with one-third its weight of coarse aluminium powder or fine borings. Use a clay crucible of

such a size that the mixture does not more than three-quarters fill it.

The ignition powder used is made up of one part aluminium powder and twelve parts of barium peroxide.

Place at first only a spoonful of the pyrolusite mixture in the crucible, and keep the rest conveniently near. Cover that which is in the crucible with a layer of ignition powder, heap the latter up slightly in the centre, and insert in it a strip of magnesium ribbon.

Light the ribbon, and the heat communicated to the ignition powder will cause a vigorous combustion, which in its turn will cause the pyrolusite mixture to react.

Keep clear of flying sparks, and gradually add the rest of the mixture without allowing the reaction at any time to cool down. When the reaction is complete and the cooled crucible contents are knocked out, a small cake of metallic manganese will be found at the bottom, weighing approximately 12 gms.

***Exp. 10.** Make a borax bead by fusing some borax in the platinum wire loop of Fig. 15*a*. When it is quite clear and glass-like, place on it a trace of pyrolusite, and fuse well. On cooling, an amethyst bead is obtained.

PIGMENTS

The following experiments are connected with the preparation of certain substances used as paints or pigments.

Exp. 11. White Pigments.

**White Lead* ($2PbCO_3$, $Pb(OH)_2$).—Fill a test-tube half full of lead acetate solution, add an equal quantity of sodium carbonate solution, and shake well. A white ppt. of basic lead carbonate is formed, which should be filtered through a small filter-paper and kept.

Zinc White.—Fill a test-tube half full of zinc sulphate solution, add an equal volume of ammonium hydroxide. Filter the white ppt. of zinc hydroxide, and keep.

Equation—

$$ZnSO_4 + 2(NH_4)OH = Zn(OH)_2 + (NH_4)_2SO_4.$$

True *zinc white* is got by ignition of this hydroxide, or by burning metallic zinc.

$$Zn(OH)_2 = \underset{\text{Zinc white.}}{ZnO} + H_2O.$$

**Barium White.*—Fill a test-tube half full of barium chloride, add an equal volume of dilute sulphuric acid. Filter the white ppt. of barium sulphate ($BaSO_4$).

Now spread the three filters containing these white pigments on the bench, and add to each one a few drops of water containing hydrogen sulphide.

The white lead becomes blackened, the zinc white slowly assumes a greyish hue, and the barium white remains unchanged. Which of these paints would retain their white colour best in a town where the air contains small amounts of sulphur gases?

Exp. 12. Yellow, Orange, and Red Pigments.

**Chrome Yellow.*—To a solution of lead acetate add potassium chromate solution. A yellow ppt. of lead chromate is produced.

Equation—

$$Pb\bar{A}_2 + K_2CrO_4 = PbCrO_4 + 2K\bar{A}.$$

Divide the yellow liquid into two parts, and add to one a few drops of soda. The yellow colour is changed to orange, *i.e.* chrome orange.

To the second portion of chrome yellow add more soda. The colour becomes red, *i.e.* chrome red.

By addition of a drop of hydrogen sulphide water, show that all these "chrome" colours become blackened.

Zinc Yellow.—To a solution of zinc sulphate add potassium chromate; a yellow ppt. of zinc chromate is formed.

Royal Yellow.—To a solution of white arsenic in dilute hydrochloric acid add hydrogen sulphide. A yellow ppt. of arsenic sulphide (As_2S_3) is formed.

Cadmium Yellow.—To a solution of cadmium sulphate add hydrogen sulphide. A pale yellow ppt. of cadmium sulphide is formed (CdS).

Antimony Orange.—To a solution of antimony chloride in

dilute hydrochloric acid add hydrogen sulphide. An orange ppt. of antimony sulphide (Sb_2S_3) is formed.

**Red Lead.*—Place a small quantity of lead oxide (litharge) on a small piece of aluminium, which acts as a tray. Support this on a pipeclay triangle, and heat by a Bunsen flame just to a dull-red heat. The yellow powder becomes changed to red, and by turning over the powder occasionally the whole may be so changed.

Exp. 13. Green and Blue Pigments.

**Prussian Blue.*—To a solution of ferric chloride add potassium ferrocyanide, and shake well. A blue ppt. is formed of Prussian blue.

Smalt.—A blue pigment formed by fusing in a crucible 1 part of cobalt oxide, 1 part silica, and 2 parts of fusion mixture.

**Brunswick Green.*—Prepare Prussian blue as above by treating ferric chloride solution with ferrocyanide of potassium.

In a second test-tube prepare barium white by adding dilute sulphuric acid to barium chloride.

In a third tube prepare chrome yellow by adding potassium chromate to lead acetate solution.

Now pour the chrome yellow into an evaporating dish, and add the Prussian blue solution.

Mix well with a glass rod, and observe the resultant green colour obtained. Filter half of this liquid, and keep the pigment on the filter-paper.

To the other half add part of the barium white, and stir with a glass rod. This gives a paler green, and by adding more barium white a still fainter shade can be obtained. Filter this sample of Brunswick green. Now place both dark and light samples of the pigment on the bench, and try the effect of adding a few drops of hydrogen sulphide water to each.

Chrome Green.—Fuse in a porcelain crucible 1 part of potassium dichromate mixed with 2 parts of boric acid, at a dull red-heat. After keeping in a fused condition for ten minutes, allow the melt to cool by placing the crucible on an iron sheet. Lixiviate the cool mass with hot water, and

finally powder up any lumps, in a mortar with warm water. Filter the extracted mass, and wash twice on the filter with warm distilled water, and dry. Spread the light-green pigment on the bench, and add a few drops of hydrogen sulphide water to it.

The colour basis in this pigment is chromic oxide (Cr_2O_3).

**Paris Green.*—This pigment is a double one, being a mixture of acetate and arsenite of copper, and is prepared as follows :—

Place a few gms. of metallic copper in a porcelain dish. Add to this a few c.cms. of acetic acid or vinegar, and warm over a small flame. Do not let the substance dry up, but add acetic acid occasionally to make up for loss by evaporation. The copper ultimately dissolves, and on drying, a green mass of basic copper acetate is obtained. This itself is used as a green pigment under the name of "verdigris." Powder the dried mass, and keep it on one side.

Now prepare some copper arsenite by adding sodium arsenite solution to a few c.cms. of copper sulphate solution in a boiling-tube. Shake well, and filter the light-green ppt. Dry this over a flame carefully, and when powdered, mix with the verdigris previously made. By this means an emerald green colour is obtained, known as "Paris green."

Add some hydrogen sulphide solution to this in a dish, and observe its change in colour.

CHAPTER XI

SIMPLE QUALITATIVE ANALYSIS

TESTS are here given which are necessary for the identification of any ordinary metal or acid. A scheme of analysis is attached, by means of which the student may analyse with accuracy any one common salt. This scheme is based upon general analytical methods, and will therefore serve for analysing complex mixtures in many cases.

A mineral substance may contain metals, non-metals, metallic oxides, acids, or salts, and in the general scheme, methods will be given for dealing with each class. The tests used, fall into two divisions—dry tests and wet tests.

The former tests are preliminary in nature, and are used chiefly for identifying metals or the metallic constituents of salts and oxides.

The most important tests are the *wet tests*, carried out in solution; and to apply these the original substance must be dissolved in either water or hydrochloric acid.

If a substance is quite insoluble, it will be treated in a special manner.

Certain metals and acids are grouped together where one particular reagent has a marked effect on them, thereby marking them off from the rest.

The members of a particular group can be separated from the rest in this way, and then the individual members of a group can be separated from each other.

THE METALS

Group I.—Metals whose Chlorides are Precipitated in the Cold by Hydrochloric Acid: Lead, Silver, Mercury (Mercurosum), Tungsten.

***Lead.** *Dry Tests.*—Use lead oxide.

1. Heated in a dry ignition-tube, the oxide darkens in colour.

2. Heated on charcoal (see p. 10) in the blowpipe flame, a metallic bead of lead is obtained by reduction, and a yellow incrustation is formed on the charcoal. The bead is soft, and marks paper.

3. Heated in the flame on a platinum wire loop moistened with strong HCl, a livid blue flame is given.

Wet Tests.—Use lead nitrate, $Pb(NO_3)_2$, dissolved in water.

1. Addition of hydrochloric acid precipitates white lead chloride, soluble in *boiling* water, from which the lead chloride deposits in glistening crystals on cooling.

$$Pb(NO_3)_2 + 2HCl = PbCl_2 + 2HNO_3.$$

2. H_2S passed into a solution acid with HCl, gives a black ppt. of lead sulphide, soluble in boiling dilute nitric acid.

$$Pb(NO_3)_2 + H_2S = PbS + 2HNO_3.$$

3. Dilute sulphuric acid precipitates white lead sulphate.

$$Pb(NO_3)_2 + H_2SO_4 = PbSO_4 + 2HNO_3.$$

Silver.—Use silver nitrate crystals.

Blowpipe Test.—Heated on charcoal in reducing flame, a soft metallic bead of silver is obtained, but no incrustation. The bead does not mark paper like lead.

Wet Tests.—Use silver nitrate solution.

1. Hydrochloric acid precipitates white silver chloride, insoluble in boiling water, but soluble in ammonia.

$$AgNO_3 + HCl = AgCl + HNO_3.$$

Mercury (Mercurosum).—Use mercurous nitrate solution.

1. Hydrochloric acid precipitates white mercurous chloride, insoluble in boiling water and ammonia, but turned black by ammonia.

$$Hg_2(NO_3)_2 + 2HCl = Hg_2Cl_2 + 2HNO_3.$$

Tungsten.—Use sodium tungstate solution.

1. Hydrochloric acid precipitates white tungstic acid, which on boiling becomes yellow. This ppt. is soluble in AmOH, but insoluble in nitric acid.

2. Add to a solution of tungstate, a piece of zinc and a few drops of dilute hydrochloric acid. The solution turns blue.

TABLE I.—*For Detection and Separation of Pb, Hg, Ag, W.*

Add to the solution a few drops of strong HNO_3 till acid White ppt. = H_2WO_4. Filter.

(*a*) Boil a small portion of ppt. with water. Turns yellow.	To filtrate, add HCl till no more ppt., and filter. ‡ Wash with cold water, and then boil residue with water, and filter.	
(*b*) Dissolve a portion in dilute AmOH. Add a piece of zinc, and then acidify with HCl. Blue colour = W.	(*a*) Cool hot filtrate under tap. Glistening white crystals of $PbCl_2$ separate. (*b*) Pour off a few drops of top liquor and add K_2CrO_4. Yellow ppt. of $PbCrO_4$.	To residue on filter add dilute AmOH, and collect filtrate. Blackening = mercurosum. Acidify the filtrate with HNO_3. A white curdy ppt. which does not turn yellow on boiling = AgCl.

Group II.—Metals whose Sulphides are Precipitated by H_2S in Presence of Hydrochloric Acid:—

Group II. (a).—Sulphides insoluble in Ammonium Sulphide: **Mercury (Mercuricum), Lead, Copper, Bismuth, Cadmium.**

Group II. (b).—Sulphides soluble in Ammonium Sulphide: **Arsenic, Antimony, Tin.**

Group II. (a). Mercury.—Use mercuric chloride.

Dry Tests—

1. Heated in an ignition-tube. A white sublimate is formed by all mercury compounds.

2. Heated in an ignition-tube with reducing mixture (fusion mixture + KCN). A mirror of mercury forms on the cooler portions of the tube. This can be rubbed to globules, and the globules tilted out on to paper.

Wet Tests.—Use an aqueous solution of mercuric chloride.

1. H_2S produces a ppt. which is finally black. This ppt. is first white, and as more H_2S passes it becomes red, and then black. It is insoluble in boiling dilute nitric acid, but soluble in aqua regia.

2. Place a bright strip of copper in a little of the solution, which has been acidified with HCl. The copper is quickly covered with a deposit of mercury.

***Copper.**—Use copper sulphate.

Dry Tests—

1. Heated on charcoal; bright red spicules of copper are formed.

2. Heated on platinum wire, a green flame is obtained.

3. Heated in a borax bead in the oxidising flame, a green bead is obtained. In the reducing flame this bead becomes either colourless or red.

Wet Tests.—Use copper sulphate solution.

1. A piece of polished iron or steel, dipped into the solution, is coated with copper.

2. H_2S passed into the solution, acidified with hydrochloric acid, gives a black ppt. of copper sulphide. Soluble in boiling dilute nitric acid.

$$CuSO_4 + H_2S = CuS + H_2SO_4.$$

3. Ammonia solution gives at first a greenish-blue ppt. of copper hydroxide, but on adding excess of ammonia the solution clears and becomes deep blue.

Bismuth. *Dry Tests.*—Use bismuth nitrate.

1. Heated on charcoal with fusion mixture, brittle white beads of metallic bismuth are formed, together with a yellow incrustation.

Wet Tests.—Use bismuth nitrate dissolved in dilute nitric acid.

1. H_2S gives a dark brown ppt. of the sulphide, soluble in boiling dilute nitric acid.

$$2Bi(NO_3)_3 + 3H_2S = Bi_2S_3 + 6HNO_3.$$

2. On pouring a few drops of the clear solution into a

beaker of water, a cloudy ppt. is formed of bismuth oxynitrate.

Cadmium. *Dry Tests.*—Use cadmium sulphate.

1. Heated on charcoal, a brown incrustation is formed.

Wet Tests.—Use cadmium sulphate solution.

1. H_2S gives a pale yellow ppt., soluble in boiling dilute nitric acid, but insoluble in ammonium sulphide.

TABLE II. (A).—*Detection and Separation of Mercuricum, Lead, Bismuth, Copper, Cadmium.*

Precipitate obtained by H_2S passed into a solution acid with HCl, and which is insoluble in warm Am_2S, may contain

$$HgS,\ PbS,\ Bi_2S_3,\ CuS,\ CdS.$$

Boil the ppt. with dilute nitric acid for a few moments, and filter.

<table>
<tr>
<td rowspan="2">Residue, black, may contain sulphur only, or sulphur and HgS. Therefore warm it in a dish with a few drops of aqua regia. Then add ammonia in slight excess, with stirring, and finally acidify with dilute hydrochloric acid.
Filter from any black residue remaining, and into the warm filtrate place a piece of bright metallic copper.
[A grey deposit = mercury.</td>
<td colspan="3">To filtrate, add a few drops of dilute H_2SO_4, and evaporate to small bulk. Filter.</td>
</tr>
<tr>
<td>White ppt. = $PbSO_4$.
This ppt. dissolves in boiling ammonium-acetate solution.</td>
<td colspan="2">Add AmOH to filtrate and stir, till strongly alkaline. Filter.</td>
</tr>
<tr>
<td rowspan="2"></td>
<td rowspan="2">White ppt., bismuth.
Dissolve it in a few drops of HCl, and pour into water.
White cloud confirms bismuth.</td>
<td>Filtrate. Deep blue shows copper.
If blue, follow (1).
If not blue (no copper), follow (2).</td>
<td></td>
</tr>
<tr>
<td>(1) Add KCN solution till colourless, and pass H_2S.
Yellow ppt. = Cd.</td>
<td>(2) Pass H_2S.
Yellow ppt. = Cd.</td>
</tr>
</table>

Group II. (B).—**Arsenic.** *Dry Tests.*—Use arsenious oxide.

1. Heated in an ignition-tube, with reducing mixture, a mirror of arsenic is formed in the cooler part of the tube, and a smell of garlic will be noticed.

2. Heated on charcoal, a white incrustation is obtained, and a garlic odour.

3. Heated on platinum wire, the flame is livid blue.

Wet Tests.—Use arsenious oxide dissolved in hydrochloric acid.

1. Marsh's test. See p. 103.

2. H_2S gives a canary-yellow ppt., soluble in warm ammonium sulphide. Insoluble in boiling strong hydrochloric acid.

Antimony. *Dry Tests.*—Use antimony oxide.

1. Heated on charcoal with fusion mixture, brittle white beads of metallic antimony result.

Wet Tests.—Use antimony oxide (Sb_2O_3) dissolved in dilute hydrochloric acid.

1. H_2S gives an orange ppt. of antimony sulphide, soluble in ammonium sulphide, and also in boiling concentrated HCl.

2. Pour a few drops of the solution into a dish containing a piece of platinum in contact with a piece of zinc. A black stain of antimony forms on the platinum.

***Tin.** *Dry Tests.*—Use tin chloride ($SnCl_2$).

1. Heated on charcoal. Residue is yellow while hot, and becomes white on cooling.

2. Heated on charcoal with fusion mixture, malleable beads of metallic tin are formed, which do not mark paper.

Wet Tests.—Use tin chloride dissolved in dilute HCl.

1. H_2S gives a brown ppt. of tin sulphide, soluble in warm ammonium sulphide, and also in boiling concentrated HCl.

$$SnCl_2 + H_2S = SnS + 2HCl.$$

2. Mercuric chloride solution gives a white ppt. of mercurous chloride. This ppt. becomes grey on boiling, if tin chloride is in excess.

TABLE II. (B).—*Detection and Separation of As, Sb, and Sn.*

Precipitate obtained by passing H_2S, which is *soluble* in Am_2S, and reprecipitated by acidifying the solution with HCl.

The ppt. is boiled in a small flask, with concentrated HCl, for a few moments, and filtered.

Yellow residue = arsenic. Confirm by mixing with four times its weight of reducing mixture, and heating in an ignition-tube, mirror of arsenic obtained.	Divide the filtrate into two portions.	
	(1) Boil with a piece of copper for five minutes. Pour off the liquid into a test-tube and add mercuric chloride solution. A white ppt., turning grey on boiling, denotes tin.	(2) Pour this on to a piece of platinum in contact with a piece of zinc. A black stain on the platinum indicates antimony. Wash the black stain, and add two drops of Am_2S. Evaporate to dryness. Orange stain = Sb.

Group III. (A).—Metals whose Hydroxides are Precipitated on addition of AmCl and AmOH: Iron, Aluminium, and Chromium.

***Iron.** *Dry Tests.*—Use ferric chloride.

1. Heated in a borax bead in oxidising flame, a yellow bead is obtained. This becomes green after heating in the reducing flame.

2. Heated on charcoal with fusion mixture, black particles of iron are obtained which are attracted by a magnet.

Wet Tests.—Use ferric chloride solution.

1. AmOH or KOH gives a reddish-brown ppt. of ferric hydroxide, insoluble in excess, but soluble in HCl.

$$3AmOH + FeCl_3 = 3AmCl + Fe(OH)_3.$$

2. Am_2S gives a black ppt. of iron sulphide, soluble in dilute HCl.

3. Potassium ferrocyanide gives a dense blue ppt. of Prussian blue.

***Aluminium.** *Dry Tests.*—Use aluminium sulphate.

1. Heated on charcoal, a white mass, very luminous when hot, is obtained. If this be cooled, and then moistened with two drops of cobalt nitrate solution, on heating again a blue mass is obtained.

Wet Tests.—Use aluminium sulphate in solution.

1. AmOH gives a white gelatinous ppt. of $Al(OH)_3$, insoluble in excess, but soluble in dilute HCl.

$$6AmOH + Al_2(SO_4)_3 = 2Al(OH)_3 + 3Am_2SO_4.$$

2. NaOH solution gives a similar white ppt., easily soluble in excess of NaOH. It may be reprecipitated from the solution by just making neutral with HCl.

Chromium. *Dry Tests.*—Use chrome alum.

1. Heated on charcoal with fusion mixture, and a little potassium nitrate, a yellow mass of chromate is obtained.

2. Heated in a borax bead, in both oxidising and reducing flames, a green bead is obtained.

Wet Tests.—Use chrome alum in solution.

1. AmOH gives a blue-green ppt. of chromium hydroxide, partly soluble in excess, giving a violet solution. It is completely reprecipitated by boiling the solution.

2. NaOH solution gives a similar ppt., soluble in excess to a green solution. Boiling completely reprecipitates the $Cr(OH)_3$.

$$CrCl_3 + 3AmOH = Cr(OH)_3 + 3AmCl.$$

TABLE III. (A).—*Detection and Separation of Fe, Al, Cr.*

Precipitate obtained by addition of AmCl and then AmOH.

Note.—The addition of AmCl is necessary before adding the AmOH. Transfer the ppt. to a beaker. Add excess of NaOH solution, boil and filter.

Filtrate may contain aluminium. Make the solution acid with HCl, and then add a slight excess of AmOH. Colourless gelatinous ppt. = $Al(OH)_3$. Confirm by fusing on charcoal, and moistening with cobalt nitrate. Blue colour = Al.	Dry the residue quickly, mix it with an equal quantity of fusion mixture and a little KNO_3, and fuse on a piece of porcelain or platinum foil until frothing ceases. Cool and then boil the residue with water, and filter.	
	Residue, red = Fe. Confirm by dissolving a little in HCl, and then add potassium ferrocyanide. Prussian blue = Fe.	Yellow filtrate = Cr. Acidify a little with acetic acid, and add lead acetate. Yellow ppt. = lead chromate.

Group III. (B).—**Metals whose Sulphides are Precipitated by Am_2S: Cobalt, Nickel, Zinc, Manganese.**

Cobalt. *Dry Tests.*—Use cobalt nitrate.

1. Heated in a clear borax bead, it gives a blue bead, both in oxidising and reducing flames.

2. Heated on charcoal with fusion mixture, it gives black particles attracted by a magnet.

Wet Tests.—Use cobalt nitrate solution.

1. Am_2S gives a black ppt. of cobalt sulphide, insoluble in *very dilute* hydrochloric acid (1 in 10).

Nickel.—The tests are the same as for cobalt. The only difference is in the borax bead.

Nickel gives a violet or reddish-brown bead in the oxidising flame, which becomes grey and opaque after heating in the reducing flame.

***Zinc.** *Dry Tests.*—Use zinc sulphate.

1. Heated on charcoal and then moistened with cobalt nitrate and reheated, gives a green mass.

2. Zinc compounds when heated give an oxide of zinc, which is yellow when hot, and white when cold.

Wet Tests.—Use zinc sulphate solution.

1. Am_2S gives a white ppt. of zinc sulphide, soluble in very dilute hydrochloric acid (1 in 10).

2. NaOH solution gives a white ppt. of zinc hydroxide, easily soluble in excess.

$$2NaOH + ZnSO_4 = Zn(OH)_2 + Na_2SO_4.$$

Passing H_2S into this solution causes a white ppt. of ZnS.

Manganese. *Dry Tests.*—Use manganese sulphate.

1. Heated in borax bead in oxidising flame, it gives an amethyst bead, which after fusing in the reducing flame becomes colourless.

2. Fused on a piece of porcelain or platinum foil with fusion mixture and potassium nitrate, a green mass is obtained.

Wet Tests.—Use manganese sulphate solution.

1. Am_2S gives a pink coloured ppt. of manganese sulphide, easily soluble in weak HCl (1 in 10).

2. NaOH precipitates white manganese hydroxide, which rapidly darkens on exposure to air.

TABLE III. (B).—*Detection and Separation of Nickel, Cobalt, Zinc, Manganese.*

Ppt. obtained by addition of Am_2S is digested in the cold for five minutes with dilute hydrochloric acid (1 part of acid to 9 parts of water). Filter.

<table>
<tr><td>Residue, black, may be NiS or CoS.
Place some of the ppt. on a borax bead, and fuse.
Blue bead in both flames = cobalt.
Yellow in oxidising and grey in reducing = nickel.

Note.—The separation of nickel and cobalt from each other is somewhat difficult, and the student who desires to effect such separation must refer to a book on analytical chemistry.</td><td colspan="2">To the boiled filtrate add NaOH till strongly alkaline. Stir and filter.</td></tr>
<tr><td></td><td>Residue white at first, but rapidly goes brown on the filter-paper = manganese.
Confirm by fusing with fusion mixture and nitre.
Green mass = manganese.</td><td>To filtrate add H_2S.
White ppt. = ZnS.
Confirm zinc by heating the ppt. on charcoal and moistening with cobalt nitrate.
Green mass = zinc.</td></tr>
</table>

Group IV.—Metals whose Carbonates are Precipitated by Am_2CO_3 in Neutral or Alkaline Solution: Barium, Strontium, Calcium.

Barium. *Dry Tests.*—Use barium chloride.

1. Heated on platinum wire gives an apple-green flame coloration.

Wet Tests.—Use barium chloride in solution.

1. H_2SO_4 gives a white ppt. of $BaSO_4$, insoluble in dilute HCl.

2. Am_2CO_3 gives a white ppt. of barium carbonate, soluble in dilute acids.

$$Am_2CO_3 + BaCl_2 = BaCO_3 + 2AmCl.$$

3. Potassium chromate gives a yellow ppt. of barium chromate, insoluble in acetic acid.

$$K_2CrO_4 + BaCl_2 = BaCrO_4 + 2KCl.$$

Strontium. *Dry Tests.*—Use strontium chloride.

1. Heated on platinum wire gives a crimson flame.

Wet Tests.—Use strontium chloride solution.

1. Am_2CO_3 gives a white ppt. of strontium carbonate, soluble in dilute acids.

2. $CaSO_4$ or H_2SO_4 gives a white ppt. of strontium sulphate on standing a short time. The precipitation is accelerated by boiling.

***Calcium.** *Dry Tests.*—Use calcium chloride.

1. Heated on platinum wire gives a brick-red flame.

Wet Tests.—Use calcium chloride solution.

1. Am_2CO_3 gives a white ppt. of calcium carbonate, soluble in dilute acids.

2. H_2SO_4 gives a white ppt. of $CaSO_4$ in strong solutions, but $CaSO_4$ solution gives no ppt.

3. Ammonium oxalate gives a white ppt. of calcium oxalate, soluble in mineral acids, but insoluble in acetic acid.

TABLE IV.—*Detection and Separation of Barium, Calcium, and Strontium.*

Ppt. produced by the addition of Am_2CO_3 in presence of AmCl may contain $BaCO_3$, $SrCO_3$, $CaCO_3$.

Dissolve it in dilute acetic acid, and to a small portion

I. Add a few drops of potassium chromate. Yellow ppt. = barium.

If barium is present, proceed by IV.

II. If barium is absent, add to a fresh portion of solution a few drops of $CaSO_4$. White ppt. at once or on boiling = strontium.

If strontium is present, proceed by IV.*

III. If barium and strontium are both absent, test a fresh portion of solution for calcium by adding H_2SO_4, and another portion by adding ammonium oxalate.

A white ppt. in both cases indicates calcium.

IV. To the solution of carbonates in dilute acetic acid, K_2CrO_4 solution is added in excess. Filter.

Yellow ppt. is $BaCrO_4$.	*To the filtrate add dilute H_2SO_4, boil and filter.	
	White ppt. is $SrSO_4$, and may contain some $CaSO_4$. Heat on platinum wire in Bunsen. Crimson confirms Sr.	To filtrate add AmOH till alkaline, and then ammonium oxalate. White ppt. = calcium oxalate.

Group V.—Magnesium, Sodium, Potassium, Ammonium.

Note.—There is no group reagent for these four radicles.

Magnesium. *Dry Tests.*—Use magnesium sulphate.

1. Heated on charcoal and then moistened with cobalt nitrate, a pink mass is obtained.

Wet Tests.—Use magnesium sulphate solution.

1. AmOH produces a white ppt. of $Mg(OH)_2$.

If AmCl solution is added first, then AmOH gives no ppt.

2. Sodium phosphate solution, added after AmCl and AmOH, gives a white crystalline ppt. of $MgAmPO_4$.

***Sodium.** *Dry Tests.*—Use sodium chloride.

1. Heated on platinum wire it gives a golden-yellow flame, which is not visible through blue glass.

There are no good "wet tests" for sodium salts.

Potassium. *Dry Tests.*—Use potassium chloride.

1. Heated on platinum wire it gives a lilac-coloured flame, which appears red though blue glass.

Wet Tests.—Use potassium chloride solution.

1. Sodium bi-tartrate, added to a neutral solution, gives a white ppt. of potassium bi-tartrate, especially after well shaking.

***Ammonium.** *Dry Tests.*—Use ammonium chloride.

1. Heated in a test-tube, ammonium salts sublime in white crystals on the cool portions of the tube.

Wet Tests.—Use ammonium chloride solution.

1. Sodium bi-tartrate gives a white ppt. in neutral solutions, especially after shaking.

2. NaOH solution added to ammonium salts in solution, causes an evolution of NH_3, especially on warming. The NH_3 is detected by its smell and action on red litmus paper.

TABLE V.—*Detection and Separation of Ammonium, Magnesium, Potassium, and Sodium.*

The ammonium radicle must be tested for in the original solution, or solid, by warming with NaOH.

Evolution of NH_3 indicates an ammonium salt.

The solution must contain the metals as chlorides $MgCl_2$, KCl, NaCl.

To one portion add AmCl, then AmOH, and finally sodium phosphate (Na_2HPO_4), and shake well.

A white crystalline ppt. = magnesium.

If magnesium is present, proceed by *A* before going to *B*.

,, ,, absent, ,, ,, *B*.

A. The magnesium is removed from solution by adding baryta water, $Ba(OH)_2$, until no further ppt. is formed. Filter and neglect the residue, $Mg(OH)_2$.

Pass CO_2 into the filtrate to remove any excess of $Ba(OH)_2$, in the form of $BaCO_3$, and when no more ppt. forms, filter and neglect residue.

B. Filtrate may contain NaCl, KCl.

Divide it into two portions.

To one portion add sodium bi-tartrate and shake well. A white ppt. = potassium.

Into the second portion dip a platinum loop, and place the loop in the Bunsen flame. A persistent golden-yellow flame indicates sodium.

If potassium is present as well, the red colour will be seen when the flame is viewed through blue glass.

Tests for Commoner Acid Radicles.

***H_2SO_4 (Sulphuric Acid).**—Use sodium or potassium sulphate.

1. $BaCl_2$ solution added to a solution of a sulphate gives a white ppt. of $BaSO_4$ insoluble in HCl.

H_2SO_3 (Sulphurous Acid).—Use sodium or potassium sulphite.

1. Addition of dilute HCl to the solid, or solution of a sulphite, produces an evolution of sulphur dioxide, SO_2, which may be detected by its smell and its power of turning chromate paper green.

2. $BaCl_2$ solution gives a white ppt. of $CaSO_3$, soluble in HCl.

***H_2CO_3 (Carbonic Acid).**—Use sodium carbonate, Na_2CO_3.

1. Dilute HCl added to the solid, or solution, produces an evolution of CO_2, recognised by its turning a drop of lime-water, on the end of a rod, milky.

***H_2S (Hydrosulphuric Acid).**—Use iron sulphide, FeS.

1. Dilute HCl added to the solid or to a solution of any sulphide, produces an evolution of H_2S, which gas is recognised by its smell, as well as by its turning a lead-paper black.

HNO_2 (Nitrous Acid).—Use sodium nitrite.

1. Dilute HCl added to the solid, or a solution, produces an evolution of reddish fumes, smelling like nitric acid, and which do not turn starch-paper orange.

***HNO_3 (Nitric Acid).**—Use potassium nitrate.

1. Concentrated H_2SO_4 added to a solid nitrate forms free HNO_3. If now a piece of copper be added, reddish nitrous fumes are evolved.

2. $FeSO_4$ solution is mixed with the nitrate solution, and then concentrated H_2SO_4 is poured carefully down the side of the tube. The H_2SO_4 runs to the bottom, and where the two liquids meet a brown ring is formed (Brown Ring Test).

***HCl (Hydrochloric Acid).**—Use sodium chloride.

1. Concentrated H_2SO_4 added to the solid, produces an evolution of HCl gas which fumes strongly in air.

2. $AgNO_3$ added to a solution of a chloride gives a white curdy ppt. of AgCl, which is insoluble in HNO_3, but soluble in AmOH.

H_3PO_4 (Phosphoric Acid). — Use sodium phosphate, Na_2HPO_4.

1. Magnesia mixture added to a solution of phosphate, produces a white ppt. of $MgNH_4PO_4$, especially after shaking.

2. Ferric chloride solution gives a yellow ppt. of ferric phosphate, in neutral solutions.

***H_2SiO_3 (Silicic Acid).**—Use sodium silicate.

1. Fused in a "microcosmic salt bead" in a platinum loop, the silicate forms a skeleton framework.

2. If a solution containing silicate is evaporated to dryness in a dish with HCl to complete dryness, a residue of SiO_2 is left, which will remain undissolved when the remainder of the residue has been extracted with HCl.

Chloric Acid.—Use potassium chlorate.

1. A chlorate when heated on charcoal, causes it to deflagrate.

2. Heated in an ignition-tube, oxygen is evolved, which rekindles a glowing splinter.

3. After strong ignition the residue from 2 answers the tests for a chloride.

Scheme of Analysis to be followed when Analysing a Salt or a Simple Mixture.

Preliminary Tests for Bases.

I. *Heat the Substance in a Hard Glass Tube.*

Observation.	Inference.
1. Substance darkens in colour.	Many metallic salts and oxides, *e.g.* Cu, and Fe salts, and Fe_2O_3, Pb_3O_4, HgO.
2. Residue yellow when hot and white when cold.	ZnO, SnO.
3. White sublimate forms.	Compounds of Hg, As (NH_4).

Note.—If one of these is suspected, confirm by the following:—

(*a*) Heat the substance in an ignition-tube, mixed with reducing mixture. A metallic mirror = As. A mirror of globules = Hg.

(*b*) Warm a little of the substance with NaOH. Smell of NH_3 confirms presence of an ammonium salt.

4. Fumes evolved turn blue litmus red.	Sulphates or chlorides.
5. Gas evolved rekindles glowing splinter.	Oxygen from chlorates, nitrates, oxides, or peroxides.

II. *Heat the Substance on Platinum Wire after Moistening with HCl, and note Colour of Flame.*

Observation.	Inference.
Blue green.	Copper.
Apple green.	Barium.
Brick red.	Calcium.
Crimson.	Strontium.
Golden yellow.	Sodium.
Lilac.	Potassium.
Livid blue.	Lead, arsenic.

III. *Heat in Borax Bead.*

Note.—This test is useless unless the substance be coloured, as colourless substances do not give any characteristic results.

Observation.	Observation.	Inference.
Oxidising Flame.	Reducing Flame.	
Yellow	Green.	Iron.
Blue green.	Colourless or red.	Copper.
Blue.	Blue.	Cobalt.
Yellow.	Grey opaque.	Nickel.
Green.	Green.	Chromium.
Amethyst.	Colourless.	Manganese.

Note.—Confirm Mn or Cr here if suspected, by fusing the substance with fusion mixture and KNO_3 on porcelain, or platinum foil.
Green mass = Mn. Yellow mass = Cr.

IV. *Heat on Charcoal, the Substance alone.*

Observation.	Inference.
Substance fuses readily and sinks into the charcoal.	Salts of alkalis and some alkaline earths.
White incrustation.	Arsenic, zinc, antimony.
Brown ,,	Cadmium, lead.
Yellow ,,	Lead.
Moisten residue with two drops of cobalt nitrate and heat again.	
Green mass.	Zinc.
Blue ,,	Phosphate, silica, alumina.
Pink ,,	Magnesium.

V. *Heat on Charcoal with Fusion Mixture and KCN.*

Observation.	Inference.
Malleable white beads.	Lead, silver, tin.
Brittle ,, ,,	Antimony, bismuth.
Reddish metal.	Copper.
Black magnetic particles.	Iron, nickel, cobalt.

Preliminary Tests for Acid Radicles.

I. *Treat the Solid Substance in a test-tube with dilute HCl.*

Observation.	Inference.
Colourless gas evolved with effervescence which turns lime-water turbid.	CO_2 from a carbonate.
Gas evolved which smells of burning sulphur, and turns chromate paper green.	SO_2 from a sulphite.
Gas evolved which smells of bad eggs and turns "lead-paper" black.	H_2S from a sulphide.
Gas with reddish colour and smelling of nitrous fumes.	Nitrous fumes from a nitrite.

II. *Treat the Solid Substance with Concentrated H_2SO_4, and Warm.*

Observation.	Inference.
1. Gas evolved which fumes in air.	HCl. from chloride.
2. Red fumes, only after adding a piece of copper.	Nitric acid from a nitrate.
3. Yellow gas smelling of chlorine, accompanied by a crackling noise.	ClO_2 from a chlorate.

Preparation of a Solution to Test for Bases.

Treat the finely powdered solid as follows, using a quantity which would well cover a penny piece.

I. Shake well in a boiling-tube with enough hot distilled water to half-fill the tube. Heat to boiling, shake well, and if any residue remains, filter. Filtrate = solution I.

Wash the residue on the filter once with boiling water, and allow the washings to run into Solution I.

Solution I. = aqueous solution.

II. If any residue remains which is quite insoluble in hot water, treat in the same manner as above with hot dilute HCl. If some insoluble substance still remains, add a few drops of concentrated HCl, and boil.

If chlorine gas is evolved at this stage, continue the boiling with concentrated HCl till chlorine ceases to be evolved, adding more HCl if necessary.

If any residue remains, filter and wash the filter once with hot dilute HCl. Filtrate = solution II.

Solution II. = hydrochloric acid solution.

Note.—Any solid which remains after the above treatment must be regarded as insoluble and dealt with as described on p. 129 for insoluble substances.

III. Solutions I. and II. should be mixed together, and any ppt. produced examined by Table I., p. 113.

If this ppt. fails to answer the tests for Ag, Pb, Hg, or W, it is probably silica, and must be tested accordingly.

The solution thus prepared may be examined according to the general table on p. 129.

Preparation of a Solution to be Used when Testing for Acid Radicles.

The original substance is boiled for fifteen minutes with a strong solution of sodium carbonate.

Sufficient of the original solid to cover a halfpenny piece is used, and the sodium carbonate must be equal to five or

six times this amount. The boiling should be conducted in an evaporating dish.

After boiling for fifteen minutes, filter, and use the filtrate to test for acids, except carbonic, as on p. 130.

All bases except sodium and potassium will be removed by this means. The following equation illustrates the case where the substance consists of copper sulphate—

$$CuSO_4 + Na_2CO_3 = \underset{\text{Residue.}}{CuCO_3} + \underset{\text{Filtrate.}}{Na_2SO_4}.$$

Examination of an Insoluble Substance.

The insoluble powder must be mixed with six times its bulk of fusion mixture in a crucible, and strongly fused for fifteen minutes. Then cool and extract with distilled water. Filter from residue, and use the clear filtrate to test for acids, as on p. 130. The residue must be dissolved in dilute nitric acid and examined for the metals (bases).

General Table for Examination of Bases in Solution.

1. To the solution add hydrochloric acid in excess, and filter. Examine the washed residue by Table I., p. 113, starting at ‡

2. Filtrate from above. Pass H_2S till saturated, and filter. Digest residual ppt. with warm Am_2S for five minutes, and filter.

Examine residue by Table II.*a*, p. 115.

Examine Am_2S filtrate by Table II.*b*, p. 116.

3. *Filtrate from H_2S ppt.*—Boil off all H_2S, and then add AmCl, and afterwards AmOH in excess.

Boil and filter. Examine residue by Table III.*a*, p. 118.

4. *Filtrate from* 3.—Add Am_2S in excess and filter. Examine residue by Table III.*b*, p. 120.

5. *Filtrate from* 4.—Warm and add Am_2CO_3 in excess. Filter. Examine residue by Table IV., p. 121.

The filtrate must be examined according to Table V., p. 123, after evaporating to dryness and igniting to remove ammon. salts.

Examination for Acid Radicles in Solution.

Add to the neutral solution, silver nitrate, and filter. Ppt. A may contain :—

Silver sulphite ,, carbonate ,, chloride ,, silicate	All white.
,, phosphate	Yellow.
,, sulphide	Black.

Solution B may contain :—

Sulphuric acid, nitrous or nitric acid, and chloric acid.

Treat part of this solution with barium chloride solution, and acidify with hydrochloric acid.

White ppt. = sulphate.

Test separately in the remainder of solution B for nitrous, nitric, and chloric acids.

Ppt. A must be treated with dilute HNO_3, and filtered. Residue white = silver chloride, soluble in AmOH.

The following substances, which may dissolve in ppt. A when treated with dilute HNO_3, evolve gases as follows :—

Sulphite	evolves	SO_2.
Carbonate	,,	CO_2.
Sulphide	,,	H_2S.

To confirm the presence or absence of phosphate or silicate, make the nitric acid solution of ppt. A just neutral by adding AmOH drop by drop, and stirring.

White ppt. gelatinous = silicate.
Yellow ppt. = phosphate.

CHAPTER XII

VOLUMETRIC METHODS OF ANALYSIS

The methods of volumetric analysis are highly accurate, and at the same time they are the most rapid of those in general use for quantitative work.

In the following exercises it will be seen how, by means of certain *standard solutions*, and assisted by certain *indicators*, it is possible to estimate the constituents of many substances quickly and with accuracy:—

Definitions—

A standard solution of any reagent is one which contains a known quantity of that reagent per litre.

A normal solution is one which contains 1 gm. equivalent of the "active reagent" per litre.

In most cases this simply means the equivalent weight in gms. of the substance used.

Weaker solutions are often required, and they may be semi-normal, deci-normal, or some suitable fractional strength of the normal solution.

Stronger solutions may be of double-normal, &c., strength.

***Exp. 1. To Prepare $\frac{N}{2}$ Sodium Carbonate Solution.**

The equivalent weight of Na_2CO_3 is one half of the molecular weight, namely, $\frac{106}{2} = 53$.

A normal solution of this reagent must therefore contain 53 gms. of Na_2CO_3 per litre, hence a $\frac{N}{2}$ solution will contain $\frac{53}{2} = 26{\cdot}5$ gms. of Na_2CO_3 per litre.

Half a litre of this will be sufficient, therefore weigh exactly 13·25 gms. of dry and pure Na_2CO_3, add it gradually to about 200 c.cms. of water in a beaker, and stir well. When the carbonate has completely dissolved, pour the solution carefully into a clean 500 c.cms. flask. Wash the beaker twice with water, and add the washings to the remainder of the solution in the flask. Finally, add water until the level reaches the mark on the neck of the flask, insert the stopper, and by inverting two or three times, thoroughly mix the contents of the flask.

Label this solution, $\frac{N}{2}$ Na_2CO_3.

*Exp. 2. To Prepare a Semi-normal Solution of Sulphuric Acid.

The equivalent weight of sulphuric acid, H_2SO_4, is one half its molecular weight, namely, $\frac{98}{2} = 49$.

A normal solution must contain 49 gms. of H_2SO_4 in the litre, and hence a semi-normal solution half that quantity, namely, 24·5 gms.

Weigh out in a small beaker between 12·5 and 13·0 gms. of strong H_2SO_4 (more than sufficient to make half a litre of $\frac{N}{2}$ H_2SO_4).

Add this to distilled water in a beaker, wash out twice, and transfer the solution to a 500 c.cms. flask. Dilute to the mark, and mix well.

This solution will be too strong, and is made so, in order that when its strength is found, it may be diluted down exactly to semi-normal strength.

Now fill a burette with this approximately $\frac{N}{2}$ acid, and clamp it in a burette stand.

Withdraw 10 c.cms. of $\frac{N}{2}$ Na_2CO_3 by means of a pipette, and run it into a small beaker or conical flask. Add to it two drops of the indicator, methyl orange, and then run the

acid from the burette carefully, until just one more drop causes the methyl orange to assume a pink tinge.

Repeat this experiment again with a fresh quantity of $\frac{N}{2}$ Na_2CO_3, and take the mean acid reading as correct.

If it requires x c.cms. of acid to neutralise 10 c.cms. of sodium carbonate, this quantity of acid solution must be made up to 10 c.cms. with water to make it exactly $\frac{N}{2}$ H_2SO_4.

From this fact it can be calculated how much water must be added to any given volume of the approximately $\frac{N}{2}$ acid, in order to make it exactly $\frac{N}{2}$.

Now withdraw a given volume of the "approximate" acid, and add to it the calculated quantity of water, mix well, and place it in the flask or a bottle, and label it $\frac{N}{2}$ H_2SO_4.

*Exp. 3. To Estimate the Strength of a Solution of Potash in Gms. per Litre.

In this case it must be remembered that the neutralisation of potash solution by sulphuric acid is represented by the equation—

$$\underset{(98\ \text{gms.})}{H_2SO_4} + \underset{(2\times 56\ \text{gms.})}{2KOH} = K_2SO_4 + 2H_2O.$$

This equation shows that 49 gms. of H_2SO_4 neutralises 56 gms. of KOH, and hence a semi-normal solution of sulphuric acid containing 24·5 gms. per litre, would neutralise $\frac{56}{2} = 28$ gms. of KOH, if 1 litre were used. We can therefore deduce that 1 c.cm. of semi-normal H_2SO_4 is equivalent to 0·028 gm. KOH.

Withdraw 10 c.cms. of the potash solution supplied, by means of a pipette, and run it into a small beaker. Add two drops of methyl orange, and then run in $\frac{N}{2}$ H_2SO_4 until the pink tinge becomes evident, stirring well during the addition.

If now the volume of $\frac{N}{2}$ H_2SO_4 used is known, the amount of KOH in 10 c.cms. taken, can be ascertained from the fact that 1 c.cm. of $\frac{N}{2}$ $H_2SO_4 = 0{\cdot}028$ gm. KOH.

Hence the weight per litre may be determined.

Exp. 4. To Estimate the Strength of AmOH Solution.

This determination is carried out in exactly the same way as that of Exp. 3.

The strength can be calculated from the fact that 1 c.cm. $\frac{N}{2}$ $H_2SO_4 = 0{\cdot}0085$ gm. NH_3.

Exp. 5. To Estimate the Strength of HCl Solution.

Place the hydrochloric acid in a burette, and run it carefully into 20 c.cms. of $\frac{N}{2}$ Na_2CO_3 solution to which two drops of methyl orange have been added.

When the mean true reading has been ascertained, the strength of the acid in gms. per litre may be calculated from the fact that 1 c.cm. $\frac{N}{2}$ $Na_2CO_3 = 0{\cdot}018$ gm. HCl, and hence 20 c.cms. $\frac{N}{2}$ $Na_2CO_3 = 0{\cdot}36$ gm. HCl.

***Exp. 6. To Estimate the Copper Present in 1 Litre of Copper Sulphate Solution.**

For this purpose a standard solution of sodium carbonate is used; 10 c.cms. of the $CuSO_4$ solution, whose content of copper is to be determined, is measured into a beaker, and a measured excess of standard Na_2CO_3 is added, and the mixed solutions are then heated to boiling-point.

The following reaction takes place, which results in the precipitation of the copper as carbonate :—

$$CuSO_4 + Na_2CO_3 = CuCO_3 + Na_2SO_4.$$

On boiling, the carbonate is converted to oxide.

The solution is next filtered from CuO, the ppt. washed twice, and the excess of Na_2CO_3 unused, is determined by adding two drops of methyl orange, and then titrating with standard sulphuric acid till the pink tinge appears.

Having ascertained how much of the original Na_2CO_3 has been used up for precipitation of the copper, it is possible to calculate the amount of copper present in the 10 c.cms. of solution which was used.

The above equation shows that 63 gms. of copper will be precipitated by 106 gms. of Na_2CO_3, hence 1 c.cm. $\frac{N}{2}$ Na_2CO_3 = 0·0157 gm. Cu.

Exp. 7. To Estimate the Zinc Present in 1 Litre of Zinc Sulphate Solution.

Use 10 c.cms. of the $ZnSO_4$ solution, and proceed in the same way as in Exp. 6.

Equation—

$$ZnSO_4 + Na_2CO_3 = ZnCO_3 + Na_2SO_4.$$

Shows that 65 gms. of zinc are precipitated by 106 gms. of Na_2CO_3, hence 1 c.cm. $\frac{N}{2}$ Na_2CO_3 = 0·01625 gm. zinc.

*Exp. 8. To Estimate Iron in a Solution of $FeSO_4$, by Permanganate.

The amount of iron in a solution or a mineral is very readily determined by the assistance of permanganate of potash, in the presence of sulphuric acid.

The standard permanganate solution is made by dissolving exactly 3·160 gms. of the solid ($K_2Mn_2O_8$) in water, in a litre flask which is filled to the mark.

When this has been done, the strength of the solution can be exactly determined against iron, by dissolving 1 gm. of fine and clean iron wire in dilute sulphuric acid in a small flask, and making the total volume up to 250 c.cms.

The iron wire used, generally contains 99·6 per cent. of iron.

When these two solutions are ready, and made as just

described, place 25 c.cms. of the iron sulphate solution in a small conical flask, and run into it, with care, standard permanganate from a burette.

Stir well during the addition, and when one drop of permanganate produces a permanent pink colour, the reaction is complete. From the volume used and the amount of iron present in 25 c.cms., namely, 0·0996 gm., the iron value of 1 c.cm. of the permanganate can be calculated. Usually 1 c.cm. of the solution made as above = 0·0056 gm. iron.

The chemical process involved is one of oxidation, in which ferrous sulphate is oxidised to ferric sulphate in the presence of sulphuric acid, thus :—

$$10FeSO_4 + K_2Mn_2O_8 + 8H_2SO_4 = 5Fe_2(SO_4)_3 + K_2SO_4 + 2MnSO_4 + 8H_2O.$$

As long as oxidation proceeds, the colour of the permanganate is discharged, and the final permanent pink denotes the completion of oxidation.

The potassium permanganate thus acts as its own indicator.

Exp. 9. To Estimate Iron in Iron Alum by Permanganate.

In iron alum, the iron is already oxidised, so that before it can be determined by $K_2Mn_2O_8$, it must be reduced to the ferrous condition. This is accomplished by warming the dissolved alum with a small piece of zinc foil and dilute H_2SO_4.

Withdraw 10 c.cms. of the ferric alum solution supplied, add to it a few c.cms. of dilute H_2SO_4 and a piece of zinc foil. Warm the flask to accelerate the process, and when the zinc has completely dissolved, cool the liquid, and titrate with standard permanganate.

If the solid iron ammonium alum is used, it will be useful, in fixing upon a suitable quantity, to remember that it contains roughly one-ninth of its weight of iron.

Exp. 10. To Estimate Iron in $FeSO_4$ Solution by Dichromate.

Standard dichromate of potassium is made by heating a small quantity of $K_2Cr_2O_7$ in a dish until it just begins to

melt, and then taking exactly 4·91 gms. of the dry powdered solid, dissolving in water, and making the volume up to 1 litre.

The solution should be standardised in exactly the same way as permanganate in Exp. 8, by means of iron solution, prepared by dissolving 1 gm. of iron wire in 250 c.cms. of dilute H_2SO_4.

In this case, since the dichromate cannot indicate its own progress, recourse is had to the blue colour given by ferrous salts with potassium ferricyanide.

The ferricyanide is placed in spots on a clean white tile, and as the dichromate runs into the titrating flask, a drop is removed occasionally on a glass rod, and added to one of the ferricyanide spots. When no blue colour is produced, the oxidation is completed. The determination of the end-point requires care, because towards the end, the blue tint fades away gradually.

If the dichromate has been made accurately as above, 1 c.cm. of the solution = 0·0056 gm. iron.

The advantage it possesses over permanganate, is that iron may be correctly estimated by it in the presence of *hydrochloric acid*, and this is not easily done when permanganate is used.

Equation—

$$6FeSO_4 + K_2Cr_2O_7 + 7H_2SO_4 = 3Fe_2(SO_4)_3 + K_2SO_4 + Cr_2(SO_4)_3 + 7H_2O.$$

Exp. 11. To Estimate Iron in Iron Alum by Dichromate.

This is carried out in the same way as in Exp. 9, with the exception that when titrating with dichromate, the end-point must be determined by spots of ferricyanide on a tile.

Exp. 12. Estimation of Chloride by Standard Silver Nitrate.

The standard silver nitrate solution used, is as a rule $\frac{N}{10}$, and is prepared by dissolving 17 gms. of $AgNO_3$ in

water, and making the volume up to 1 litre; 250 c.cms. will suffice for ordinary purposes, and in this case 4·25 gms. of $AgNO_3$ must be used.

To estimate the amount of chlorine, in gms. per litre, contained in a solution of sodium chloride, measure out 10 c.cms. of the solution into a small conical flask, add two drops of potassium chromate, and then run in the silver nitrate solution from the burette.

The following reaction takes place—

$$AgNO_3 + NaCl = AgCl + NaNO_3,$$

and a white ppt. of silver chloride is formed.

When the precipitation of the chlorine is completed, the next drop of $AgNO_3$ added, produces in the flask a pink coloration, due to the presence of red silver chromate, Ag_2CrO_4, and this indicates the end-point of the reaction.

The above equation shows that 170 gms. of $AgNO_3$ will ppt. 35·5 gms. of chlorine, and hence 1 c.cm. of $\frac{N}{10}$ $AgNO_3$ will ppt. 0·00355 gm. of chlorine.

From these facts the amount of chlorine in a solution of sodium chloride can be estimated.

*Exp. 13. To Estimate the Na_2CO_3 Content of Washing Soda.

Washing soda crystals contain, besides sodium carbonate, a considerable quantity of water of crystallisation.

Powder quickly, a few crystals of washing soda in a mortar, and weigh two separate quantities of the powder in two watch-glasses, taking between 1 and 2 gms. in each case.

Dissolve each in a separate flask or beaker, and titrate each with $\frac{N}{2}$ sulphuric acid, using two drops of methyl orange as an indicator.

Knowing that 1 c.cm. of $\frac{N}{2}$ H_2SO_4 is equivalent to 0·0265 gm. Na_2CO_3, calculate the amount of this compound in the sample used, stating the result as a percentage. The two independent results should not disagree by more than 0·5 per cent.

***Exp. 14. To Estimate the Percentage of Chalk in a Mixture of Chalk and Sand.**

Weigh accurately between 1 and 2 gms. of the mixture, place it in a beaker, and stir well with 50 c.cms. $\frac{N}{2}$ HCl. When all effervescence has ceased, filter the liquid, and wash the residue on the filter-paper twice with hot water. Now add two drops of methyl orange to the filtrate, and titrate with $\frac{N}{2}$ NaOH, the excess of HCl present.

From this result, the quantity of acid used in dissolving the chalk can be found, and knowing that 1 c.cm. of $\frac{N}{2}$ HCl is equivalent to 0·025 gm. $CaCO_3$, the percentage present in the mixture can be deduced.

Equation—

$$CaCO_3 + 2HCl = CaCl_2 + H_2O + CO_2.$$

CHAPTER XIII

QUANTITATIVE ANALYSIS FOR ENGINEERING STUDENTS

To Estimate Iron in Iron Ore.

POWDER up the ore, and take from the finely powdered sample about 2·5 gms. accurately weighed.

Digest this well in a beaker with hydrochloric acid (1 : 1) for about half an hour. Decant at the end of this time through a filter-paper, and further digest the residue with fresh acid. Test the solution by taking one drop on a glass rod and bringing it into contact with a drop of thiocyanate[1] on a white tile. If no red colour is given, the first digestion with acid has effectually extracted all iron. If, however, a red colour shows, more iron is being dissolved. The extraction process and decantation must be repeated, till the last solution gives no red colour with thiocyanate. The undissolved residue consists of silica and gangue.

The solution which contains the iron is transferred to a 250 c.cms. measuring flask, and the volume made up to the mark with cold distilled water.

Small quantities of this stock solution are withdrawn (usually 25 c.cms.); reduced by boiling gently with a scrap of zinc, as explained (p. 136), and then each reduced specimen is titrated with standard dichromate, and results calculated as shown on p. 137.

*Estimation of Sulphur and Silicon in Cast Iron.

Weigh out accurately about 5 gms. of the turnings, and dissolve them in aqua regia. If sulphur separates, it may

[1] A solution of ammonium or potassium thiocyanate is used.

be dissolved by the addition of a small fragment of potassium chlorate.

When the reaction has finished, transfer to a porcelain dish, and evaporate to complete dryness on water-bath.

When dry, place the dish in an air-oven, and gradually raise the temperature to 200°. This destroys organic matter, which becomes volatilised, and converts the siliceous matter into an insoluble form. Remove the dish from the air-oven and moisten with strong HCl. Dilute with water to about 100 c.cms., and filter the hot liquid to remove siliceous matter, SiO_2.

From the weight of SiO_2 obtained after ignition, the percentage of silicon in the specimen can be calculated.

The silica must be transferred to a crucible, and the filter burnt as described on p. 13.

2. *The filtrate* contains the sulphur in the form of sulphuric acid. It is heated to boiling, and then a dilute solution of barium chloride is added, also boiling.

When it is certain that barium chloride has been added in excess, the ppt. of $BaSO_4$ is filtered, washed, and weighed after ignition in a porcelain crucible.

Dry Assay of Galena for Lead.

The ore is powdered, and melted with metallic iron and a flux to assist melting. The reaction taking place is

$$PbS + Fe = FeS + Pb.$$

The button of metallic lead remaining behind is weighed after being cleaned.

Process.—Weigh 25 gms. of finely powdered ore, and mix it well with finely powdered sodium carbonate, 20 gms., argol (black flux), 2 gms., and borax, 2 gms., each well powdered, and transfer the mixture to a clay crucible (which has been warmed near the furnace). Bend a piece of iron rod to a U shape, so that its height is the same as the depth of the crucible, and place it in the mixture, free ends downwards. Cover the mixture with 5 gms. of dry, powdered sodium carbonate. Place the crucible in a dull red-hot muffle, and cover it with the lid. Raise the temperature gradually, and

at the end of ten minutes remove the muffle door, and stir the mixture in the crucible by grasping the iron **U** with the tongs. After another ten minutes stir again, and tap the tongs sharply while holding the iron stirrer, so that any adherent lead globules may fall into the crucible. Heat strongly for ten minutes more, then remove the iron stirrer and raise the temperature, so that the slag becomes quite fluid.

Next, remove the crucible with tongs from the furnace, and, quickly inverting, pour the fused mass into a smooth iron mould. When cool, turn the mass out of the mould and hammer the button of lead found at the bottom, so as to detach any adhering slag. Finally, clean the button by rubbing well with a hard brush, and weigh it. From the weight obtained calculate the percentage of lead in the galena used. (Reserve the lead button.)

Note.—Lead, in red lead, can be estimated in a similar manner by using 40 gms. red lead, 30 gms. Na_2CO_3, 10 gms. borax, and 10 gms. flour, well mixed together.
In this case the iron **U** is not necessary.

Assay of Lead for Silver.

The button of lead obtained in the last assay may be used. The process involves the oxidation of the lead by heating it on a shallow bone-ash cupel; part of the lead oxide is removed by the draught, and the rest sinks into the cupel. The small globule of silver remaining is weighed.

Process:

To make a Cupel.—Finely powdered bone-ash is mixed in a mortar with sufficient water to make a thick paste, which sticks together well when pressed in the fingers.

The moist paste is placed in a cupel-mould and moulded to the desired shape. It is then detached from the mould and dried by gently heating.

Note.—Freshly made cupels should not be used. It is necessary that they should be a fortnight old or more.

Cupellation of Lead Button.—Heat one of the cupels in the muffle to bright redness for ten minutes, in order to drive off all moisture.

Drop the button of lead on to the bright-red cupel by means of the tongs, and shut the door.

A black crust forms on the lead at first, but soon disappears, leaving the button brighter than the cupel. If it does not clear, raise the temperature, and drop a little powdered charcoal on it wrapped in tissue paper.

When "clearing" has taken place, reduce the draught, and let a little air enter by the muffle door. This will lower the temperature somewhat, and it need not be raised again to its original magnitude till near the end of the process. The end is near when the thin film of lead oxide shows iridescence, and soon after this the globule of silver glows out and solidifies.

Remove the cupel and let it cool. Then remove the silver globule, hammer it out on a small anvil, and clean it with a hard brush. Weigh the silver, and express the amount present in ounces per ton of lead.

$$1 \text{ ton} = 2240 \times 16 \text{ ozs.}$$

*Estimation of Sulphur in Coal.

The sulphur in coal exists in two forms: (*a*) calcium sulphate, (*b*) pyrites or iron sulphide.

To estimate total sulphur, weigh accurately about 2 gms. of finely powdered coal; and mix it in a porcelain or nickel crucible with four times its weight of dry sodium carbonate. Cover the crucible with the lid, and heat gently at first to volatilise hydrocarbons. Then gradually raise the temperature to red heat, and continue heating till the mass is white and all carbon has disappeared.

Time required for this is about one hour. When cold, add water, and filter. Wash out the crucible several times, and wash the residue on the filter-paper also. Then add a few drops of bromine water to the clear solution, and heat to boiling. The solution should now be quite colourless again. Acidify with a few drops of hydrochloric acid, heat to boiling, and add boiling barium chloride solution in excess. Remove the flame, let the white ppt. of $BaSO_4$ settle, and then filter. Wash the ppt. well by decantation, and also on the filter-paper (see p. 8), then dry in the steam-oven, and transfer the

ppt. to a weighed crucible. Burn the filter-paper in a piece of platinum wire and add the ash to the crucible. Drop on the ash one drop of HCl, followed by one drop of H_2SO_4. Then ignite the crucible strongly, and weigh the $BaSO_4$ formed. The weight of $BaSO_4$ obtained, multiplied by 0·1375, gives the weight of sulphur in the ppt.

Sulphur by Using Bomb Calorimeter Residue. (See p. 151.)

The sulphur in the fuel will be burnt to sulphuric acid, and this may be estimated as follows :—

Rinse the contents of the bomb into an evaporating dish, and evaporate to dryness after adding 5 c.cms. of concentrated hydrochloric acid. This is done in order to remove nitric acid formed by the combustion of nitrogen in the coal. Treat the dry residue with 100 c.cms. of distilled water, and add 2 c.cms. of strong hydrochloric acid.

Filter from any insoluble portion, and wash the filter twice with boiling water. Boil the solution in a beaker, and then add boiling barium chloride solution in excess.

Remove the flame, and allow the ppt. of barium sulphate to settle. If the reagent has been added in excess, one more drop of barium chloride will give no ppt. in the clear liquid.

The remainder of the process is similar to that described above.

Water Analysis for Engineers.

The chief concern for the engineer in this instance, is the suitability of a water for boiler use. All natural waters contain substances either dissolved or in suspension, and the amount of dissolved solids and their nature must be known before the quality of the water is decided. The action of dissolved salts on the boiler is twofold. Substances like carbonates of lime and magnesia act as scale-formers, and are objectionable on that account, as is the presence of sulphate of lime. Other substances do not form scale, but are dangerous, because at the steam temperature they are capable of attacking and destroying the boiler coat and fittings, thus leading to leakage. Such substances in solu-

tion are the chlorides, sulphates, and nitrates of lime, magnesia, or soda.

The total solids in solution may vary from 5 parts to 50 parts per 100,000 parts of water, in average waters.

The more pure the water, the better for boiler use, and rain water, on account of its entire freedom from dissolved solids, is the ideal water for this purpose. Rain water only contains in solution air and traces of carbon dioxide, absorbed during its passage through the atmosphere. Rain which has fallen during a thunderstorm contains also traces of nitric acid, this latter being formed by the action of the electric discharge on the nitrogen and oxygen in the air.

In most cases the cost of "catching" and storing rain water prohibits its use, and therefore an available and cheap water is taken, and, if necessary, this must be *softened* before it is used in the boiler.

Oils and fats are dangerous in water for boilers, because at steam temperature, they are likely to be converted into organic acids which will attack the fittings and lead to leakage.

See Table XX. for analyses of various natural waters.

The following analysis will supply a good idea of the nature of the water and its method of treatment for boiler use.

The determinations made are :—

1. Acidity or alkalinity.
2. Total suspended solids.
3. Total dissolved solids.
4. Temporary hardness.
5. Permanent hardness.
6. Oils and fats.

Acidity or Alkalinity.—For these determinations we use deci-normal sodium carbonate solution or deci-normal hydrochloric acid. These solutions are made and standardised as explained on p. 131.

To find out whether the water is acid or alkaline, place 50 c.cms. in a beaker, and add 2 drops of methyl orange. If the indicator is turned pink the water is acid, while if it remains an orange tint, the water is alkaline or neutral.

Repeat this test, taking a fresh beaker containing 50 c.cms. of the water, and add 2 drops of phenolphthalein. If the water is alkaline the indicator becomes pink, while if it is acid or neutral the indicator remains colourless.

To estimate total alkalinity, measure out 200 c.cms. of the water (filtered from suspended matter if necessary), add 2 drops of methyl orange, and then run in $\frac{N}{10}$ HCl from a burette. The alkalinity can be expressed in terms of calcium carbonate.

1 c.cm. of $\frac{N}{10}$ HCl is equivalent to 0·005 gm. $CaCO_3$.

The result multiplied by 500 gives the quantity per 100,000 parts of water.

To estimate total acidity, 200 c.cms of the water are taken as above, 2 drops of phenolphthalein added, and $\frac{N}{10}$ Na_2CO_3 added from a burette, until the pink colour just appears.

1 c.cm. of $\frac{N}{10}$ Na_2CO_3 is equivalent to 0·003 gm. of carbonic acid, and the acidity may be expressed in terms of carbonic acid.

Equation—

$$Na_2CO_3 + H_2CO_3 = 2NaHCO_3.$$

**Suspended Solid Matter.*—A small fluted filter-paper (diameter = 6 to 7 cms.) is dried by heating in the air-oven to 110° for one hour in a tube (see p. 18).

The dried and weighed fluted-filter is then placed in a small funnel and 200 c.cms. of the water run through.

The filter and its residue are finally washed with cold distilled water, and the filter and contents then dried in the air-oven. When fairly dry it is replaced in the filter drying-tube, and the desiccation concluded.

The gain in weight of the filter multiplied by 500, gives parts of suspended matter per 100,000 parts of water.

**Total Solids and Organic Matter.*—The water, well shaken, is measured out into a 250 c.cms. measuring flask, and filled up to the mark. This quantity is transferred gradually to a weighed platinum dish, which holds about 60 to 70 c.cms., and which is heated on a water-bath. As the water in the dish evaporates, a fresh supply is added from the measuring flask until it is emptied. The flask should be washed twice

with small quantities of distilled water, and the washings transferred to the platinum dish.

When the dish is dry, remove it from the water-bath, wipe the bottom of the dish, and place it in the air-oven to dry for one hour at 115° C. At the end of this time put it in the desiccator, before weighing.

The gain in weight represents total solids, and this multiplied by 400 gives parts per 100,000 of water.

Organic matter.—If it is desired to know the amount of organic matter, the dish and residue are heated on a triangle to redness, for five minutes. Organic matter chars under such treatment, and is completely volatilised.

When the dish is cool, add a few drops of ammonium carbonate solution, and evaporate by gentle heat, and finally vaporize any solid Am_2CO_3 residue. Do not heat too strongly, or the carbonates of lime and magnesia will be changed to oxides.

Equations—

$$MgO + (NH_4)_2CO_3 = MgCO_3 + 2NH_3 + H_2O.$$
$$MgCO_3 + \text{heat} = MgO + CO_2.$$

For this reason the bottom of the dish should never reach dull red-heat.

Transfer the dish to a desiccator to cool, and then weigh. Loss on ignition represents organic matter in 250 c.cms. This weight multiplied by 400 gives organic matter per 100,000 parts of water.

Estimation of Hardness.—Two methods are given. The first and older method gives the temporary and permanent hardness with reference to a standard soap solution.

The second method gives these quantities in terms of $CaCO_3$ by a volumetric method, using standard acid and alkali.

***Method I.**—The standard soap solution is used (see p. 163), and it is standardised before use by titrating it against standard calcium chloride made as follows:—

Dissolve 0·2 gm. Iceland spar, or finely powdered marble, in a little dilute hydrochloric acid in a porcelain dish, covered with a clock glass. When the evolution of CO_2 has ceased, remove the clock glass and wash anything adhering to it into the dish, and evaporate the contents to

dryness on a water-bath. Moisten with distilled water, and again evaporate to dryness to ensure complete removal of hydrochloric acid.

Equation—

$$CaCO_3 + 2HCl = CaCl_2 + H_2O + CO_2.$$

The residue is dissolved in water and diluted to 1000 c.cms. Transfer 50 c.cms. of this standard calcium chloride solution to a 250 c.cms. stoppered bottle, and run in soap solution 1 c.cm. at a time from a burette. Put the stopper in and shake well after each addition. If the soap solution is correct, it should require 14·25 c.cms. of this solution to give a permanent lather. That is, one which remains for three minutes. For details of preparing soap solution and standardising exactly, see p. 163.

Total Hardness.—Measure out 50 c.cms. of the sample of filtered water in the stoppered shaking-bottle, and add the soap solution from a burette until, on shaking, a permanent lather is obtained. If the amount of soap solution required is greater than 16 c.cms., take some suitable fraction of 50 c.cms. of the water for titration, say 25 c.cms., or even 10 c.cms. if the water is very hard. If this is done the volume must be made up to 50 c.cms. with distilled water.

From the table below, the corresponding amount of $CaCO_3$ per 100,000 parts of water can be read off, when the number of c.cms. of soap solution used is known. If 25 c.cms. or 10 c.cms. only have been used, as above, the numbers in the table must be multiplied respectively by 2 and by 5 to give the correct hardness.

Permanent Hardness.—Transfer 200 c.cms. of the water to a flask, and boil gently for half an hour. This will remove temporary hardness. Cool slightly, and pour the cool water through a small filter into a 200 c.cms. flask. Wash the filter with cold distilled water, and make up to 200 c.cms. Remove 50 c.cms. of this water, and estimate its hardness by soap solution as above.

The difference between this number and the total hardness gives the temporary hardness.

Table of Hardness.

Column I. represents soap solution readings.
Column II. represents amount of $CaCO_3$ in 100,000 parts of water.

1	2	1	2	1	2	1	2	1	2	1	2
c.c.		c.c.		c.c.		c.c.		c.c.		c.c.	
0·7	·00	3·3	3·64	5·9	7·29	8·5	11·05	11·1	15·00	13·7	19·13
·8	·16	·4	·77	6·0	·43	·6	·20	·2	·16	·8	·29
·9	·16	·5	·90	·1	·57	·7	·35	·3	·32	·9	·44
1·0	·48	·6	4·03	·2	·71	·8	·50	·4	·48	14·0	·60
·1	·63	·7	·16	·3	·86	·9	·65	·5	·63	·1	·76
·2	·79	·8	·29	·4	8·00	9·0	·80	·6	·79	·2	·92
·3	·95	·9	·43	·5	·14	·1	·95	·7	·95	·3	20·08
·4	1·11	4·0	5·7	·6	·29	·2	12·11	·8	16·11	·4	·24
·5	·27	·1	·71	·7	·43	·3	·26	·9	·27	·5	·40
·6	·43	·2	·86	·8	·57	·4	·41	12·0	·43	·6	·56
·7	·56	·3	6·00	·9	·71	·5	·56	·1	·59	·7	·71
·8	·69	·4	·14	7·0	·86	·6	·71	·2	·75	·8	·87
·9	·82	·5	·29	·1	9·00	·7	·86	·3	·90	·9	21·03
2·0	·95	·6	·43	·2	·14	·8	13·01	·4	17·06	15·0	·19
·1	2·08	·7	·57	·3	·29	·9	·16	·5	·22	·1	·35
·2	·21	·8	·71	·4	·43	10·0	·31	·6	·38	·2	·51
·3	·34	·9	·86	·5	·57	·1	·46	·7	·54	·3	·68
·4	·47	5·0	6·0	·6	·71	·2	·61	·8	·70	·4	·85
·5	·60	·1	·14	·7	·86	·3	·76	·9	·86	·5	22·02
·6	·73	·2	·29	·8	10·00	·4	·91	13·0	18·02	·6	·18
·7	·86	·3	·43	·9	·15	·5	14·06	·1	·17	·7	·35
·8	·99	·4	·57	8·0	·30	·6	·21	·2	·33	·8	·52
·9	3·12	·5	·71	·1	·45	·7	·37	·3	·49	·9	·69
3·0	·25	·6	·86	·2	·60	·8	·52	·4	·65	16·0	·86
·1	·38	·7	7·00	·3	·75	·9	·68	·5	·81	...	...
·2	·51	·8	·14	·4	·90	11·0	·84	·6	·97	...	...

Hardness by Standard Acid and Alkali (Method II.).

Temporary Hardness.—Transfer 200 c.cms. of the water to a flask, and boil for half an hour. Cool and then filter through a small filter-paper. Keep the filtrate for estimating permanent hardness, and add to it the washings from the filter. Now pour through the filter, placed in the neck of a conical flask, 25 c.cms. of $\frac{N}{10}$ hydrochloric acid, and wash the

filter twice with distilled water. The acid will dissolve the carbonates on the filter, thereby becoming partially neutralised. Make the filtrate up to 50 c.cms., and remove half at a time for titration with $\frac{N}{10}$ sodium carbonate and methyl orange.

Every 1 c.cm. of $\frac{N}{10}$ HCl used up on the filter, corresponds to 0·005 gm. $CaCO_3$. This number, multiplied by 500, gives the temporary hardness, in terms of $CaCO_3$ per 100,000 parts.

Permanent Hardness.—The filtrate from above will contain salts, like magnesium chloride, which produce permanent hardness. Add to the solution 25 c.cms. of $\frac{N}{10}$ sodium carbonate solution, and evaporate the whole to dryness in a platinum dish. This treatment converts lime and magnesia into insoluble carbonates ($MgCl_2 + Na_2CO_3 = 2NaCl + MgCO_3$), and also neutralises any acids giving hardness in the original liquid.

Extract the residue in the dish with warm distilled water, filter, and make up the filtrate to 50 c.cms. Use half of this at a time for titration with $\frac{N}{10}$ hydrochloric acid.

Each 1 c.cm. of the original 25 c.cms. Na_2CO_3 used up is equivalent to 0·005 $CaCO_3$. This expresses the permanent hardness in terms of $CaCO_3$, and multiplied by 500 gives the amount per 100,000 parts of water.

Temporary + permanent hardness, as above determined, equals total hardness.

Estimation of Oils and Fats in the Water.

250 c.cms. of the water is shaken up in a stoppered funnel with 25 c.cms. of petroleum ether. Let the funnel stand in an upright position for ten minutes, then run off the water layer through the tap, and tip the ethereal solution which remains behind, into a weighed porcelain dish. Wash out the funnel twice with small quantities of petroleum ether, and transfer the washings to the dish. Now place the dish on a water-bath of hot water, *with no flame underneath.* The

ether will evaporate rapidly, leaving the oil and fat behind. After drying the basin and its contents in the hot-water oven for half an hour, weigh the dish. Multiply the result by 400 to get the oil per 100,000 parts.

Estimation of Chlorides.

If a water contains above 10 parts NaCl per 100,000 parts of water, it is unfit for boiler use, because the chlorides will attack the boiler fittings.

For this estimation, 50 c.cms. of the water is placed in a conical flask, and titrated with $\frac{N}{100}$ $AgNO_3$, using K_2CrO_4 as indicator. (See p. 137 for particulars.)

If the chlorine is high, $\frac{N}{10}$ $AgNO_3$ may be used instead.

1 c.cm. of $\frac{N}{10}$ $AgNO_3$ is equivalent to 0·0058 gm. NaCl.

1 c.cm. of $\frac{N}{100}$ $AgNO_3$ is equivalent to 0·00058 gm. NaCl.

Calorific Value of Fuel by Bomb Calorimeter.

The parts of this calorimeter are indicated in the accompanying figures. Fig. 52 shows the bomb itself, which is made up of a gun-metal cylinder A, to which the cover B, can be screwed down. The cylinder is lined with gold, platinum, or enamel, to protect it from the action of acids, produced during the combustion. The milled-head screw C, works a valve for controlling the inlet of oxygen gas, and D, is an insulated wire, terminating inside the bomb.

Lead wire is used to make a gas-tight joint between the cylinder and cover.

The combustion of the fuel is carried out in oxygen gas under a pressure of between 200 and 400 lbs.

Fig. 53 shows the calorimeter itself, and the stirring apparatus. The inner portion of the calorimeter is surrounded by a water jacket and an air-space. The bomb fits into the inner vessel, and is itself surrounded by the stirring apparatus. The stirrer can be revolved by means of the wooden knob K.

Fig. 54 shows the cover alone with its accompanying fuse-wire and platinum dish. The weight of fuel used for a determination is between 0·4 and 1·0 gm., and before burning, it should be dried by heating in an air-oven to 200° C. for half an hour.

After weighing accurately in the platinum dish, the latter, with its contents, is placed on the holder, and the ignition-tube is lowered so that it just dips beneath the surface of the fuel. Ten c.cms. of water are placed in the bomb, and the

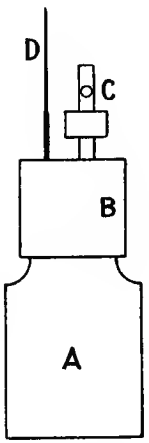

Fig. 52.

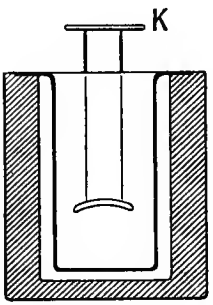

Fig. 53.

cover is then bolted on, care being taken not to jolt the bomb during the process.

The 10 c.cms. of water are added in order to absorb the acid products of combustion.

In Fig. 55, the connection between the oxygen cylinder and the bomb is shown. The gas must not be admitted too rapidly, or the powdered fuel may be blown out of the basin. For burning 0·5 gm. a pressure of 220 lbs. is sufficient, but for 1·0 gm. a pressure of 400 lbs. is necessary. When the desired pressure is reached, the screw-valve is shut, the supply tube is disconnected, and the top opening closed by a screw-nut.

The bomb is next transferred to the calorimeter, which has previously been filled with 2500 c.cms. of water, at laboratory temperature.

The terminals of an eight volt battery are connected to the bomb terminals for three seconds, in order to fire the fuel. The stirrer is now rotated, and within half a minute the thermometer will begin to rise. Maintain the stirrer in motion until no further temperature rise is noted, and make

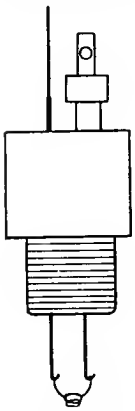

FIG. 54.

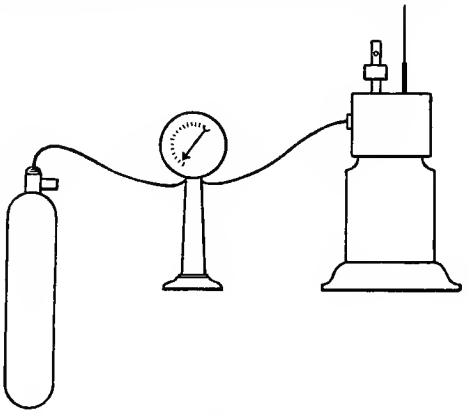

FIG. 55.

a note of the exact number of degrees and fractions, which represents the complete increase in temperature. This should be registered to the second decimal figure. If t = rise in temperature produced in 2500 c.cms. of water, and x = calorimeter equivalent, then calorific power $C = \frac{(2500+x)t}{w}$ where w = weight of fuel taken.

The value of x must be first determined by using a fuel of known calorific power. Those used are—

Naphthalene = 9692 cals.
Amorphous carbon = 8137 cals.

For the estimation of sulphur in fuel, using the oxidation product from the bomb calorimeter, see p. 144.

Analysis of Furnace Gases.

1. It is frequently desirable to know the composition of gaseous mixtures which are being used as fuel.

Such are producer-gas, water-gas, blast-furnace gas, &c., and their formation may be advantageously controlled, to increase their fuel value, when the constituent gases are quantitatively determined.

For the analyses of various furnace gases, see Table XVI.

2. Analyses of the exit gases of a furnace, afford a real check upon the process of combustion.

The gases which are heat producers, and which are burnt during the combustion, are chiefly hydrogen, hydrocarbons, and carbon monoxide. If the furnace is working properly, the exit gases should contain practically none of these. They should consist of carbon dioxide, nitrogen, and oxygen. For analyses of exit gases, see Table XVII.

These analyses are very conveniently carried out in Orsat's gas apparatus, the use of which is described here.

Use of Orsat's Apparatus for Exit Gases.

Neither soot nor moisture are estimated under ordinary conditions, and, as a matter of fact, both of these constituents condense on the sides of the collecting vessels, before the actual analysis is carried out.

A diagram of the apparatus is shown in Fig. 56.

The gas in the sampling vessel is transferred to the measuring tube A, by means of the tap and tube at B, which is connected to the sampler by thick-walled rubber tube.

The transfer of gas is accomplished by lowering the mercury or water reservoir, after the air has all been expelled from the connecting-tube, through K. The tap of the measuring tube is then opened, and 100 c.cms. of the gas to be examined, is drawn in. The tap at L is now closed, and the gas is allowed to remain in A, under slight pressure, for two or three minutes. This allows further deposition of moisture and sulphurous gases, and when the levels are readjusted, any diminution in volume should be registered.

Connection is now made between A, and C which contains

caustic potash, and the gas is passed into C and out again three times. Loss in volume = CO_2.

Oxygen is next absorbed in the alkaline pyrogallate bulb D. Lastly, the combustible gases in 50 c.cms. of the residue (which should be about 80 c.cms.), are mixed with 50 c.cms. of air and exploded in the pipette E.

After explosion, the cooled gases are returned to the measuring cylinder and the diminution in volume read off. The gases are then passed into C to absorb CO_2. Half this absorbed volume equals loss due to oxygen used in burning

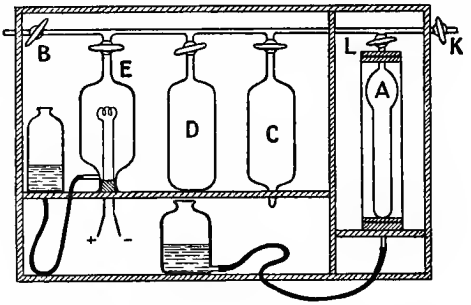

FIG. 56.

the CO, and the volume of CO_2 absorbed equals the original CO content.

$$\underset{1\text{ vol.}}{CO} + \underset{\frac{1}{2}\text{ vol.}}{O} = \underset{1\text{ vol.}}{CO_2}.$$

The remaining diminution is due to water formation when the hydrogen is burned—

$$\underset{1\text{ vol.}}{H_2} + \underset{\frac{1}{2}\text{ vol.}}{O} = \underset{1\text{ vol.}}{H_2O}.$$

Hence two-thirds of this gives the original hydrogen.

Use of Orsat's Apparatus for Furnace Gases.

A modified form of the last apparatus is used, in which there is another bulb containing cuprous chloride solution,

for directly absorbing and estimating carbon monoxide. This allows the estimation of hydrogen and methane by explosion in the pipette.

For this analysis the following order is observed in analysing 100 c.cms. of the gas :—

1. Estimate CO_2 by absorbing it in the potash bulb.
2. Estimate O_2 by absorbing it in the alkaline pyrogallate bulb.
3. Estimate CO by absorbing it in the cuprous chloride bulb.
4. Mix 50 c.cms. of the residual volume with 50 c.cms. of air, transfer to the explosion pipette, and explode.

These reactions take place with loss of volume—

$$(a) \qquad CH_4 + 2O_2 = CO_2 + 2H_2O.$$
$$(b) \qquad 2H_2 + O_2 = 2H_2O.$$

5. Measure diminution in volume, and then determine the volume of CO_2 produced by reaction (*a*).

This equals original volume of methane (CH_4), and twice this equals diminution due to methane combustion.

The remaining loss is due to reaction (*b*), the combustion of hydrogen.

Two-thirds of the latter diminution equals hydrogen.

6. Subtract the sum of (hydrogen + methane) from the total volume left after CO absorption, and count this as nitrogen.

*Analysis of Fire-Clay and Cement.

Cements.—These mixtures in the dry state consist mainly of lime and silica, together with a small quantity of alumina. The setting process depends upon *hydration*, which takes place when the powders are mixed with water. The composition of cement may be represented as that of a silicate of lime, namely, Ca_3SiO_5.

This in the presence of water becomes hydrated to an exceedingly hard mass, the following chemical change taking place—

$$2Ca_3SiO_5 + 9H_2O = Ca_2(SiO_3)_2, 5H_2O + 4Ca(OH)_2.$$

Portland cement is prepared by heating to the sintering

point, a mixture of clay with one of the following: limestone, marl, chalk, or hydraulic lime.

The tests to which cements are subjected consist of physical tests and a chemical analysis.

Physical Tests.—1. To distinguish between genuine Portland cement and a blast-furnace slag cement.

(*a*) Boil 50 gms. of the finely powdered sample with 100 c.cms. of distilled water for three hours. Keep the volume of water up, by addition of fresh quantities, and agitate occasionally to prevent the formation of lumps.

At the end of the time, filter and wash the residue with hot water, and then dry it at 110°–120° C.

When dry, determine the loss on ignition of about 1 gm.; this equals water of hydration.

Portland cement shows on an average a loss of 11·5 per cent.

Slag cement shows on an average a loss of 0·8 per cent.

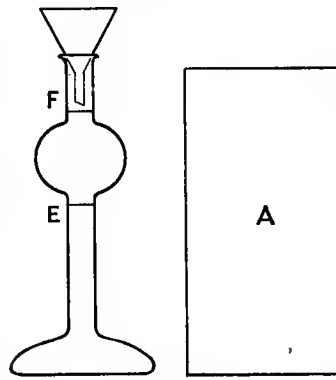

FIG. 57.

(*b*) Shake 1 to 1·5 gms. of the cement with 3 litres of freshly boiled water. Collect, and weigh the residue after ignition to determine what percentage has dissolved.

From Portland cement an average of 37 per cent. dissolves.
,, slag ,, ,, 2·3 ,, ,,

Specific Gravity.—This property is usually determined by the aid of a measuring vessel (Fig. 57), known as Le Chatelier's flask.

The finely powdered cement is dried at 100° and cooled, and 64 gms. exactly weighed out.

This quantity is transferred by means of the funnel to the flask.

This flask, which holds 120 c.cms. up to the mark F, has a neck 20 cms. long, in the middle of which is a bulb, and above and below the bulb are marks F and E.

The volume between these marks is 20 c.cms. (1·22 cubic inches). The neck has a diameter of about 9 mm., and is graduated in tenths above F.

Before introducing the cement, the flask is filled to the mark E with benzine or kerosene, and from the rise in level which results on adding the cement, its volume can be estimated. The specific gravity is obtained when the mass is divided by the volume $= 64/v$.

During the operation the flask is immersed in cold water contained in the jar A.

*Chemical Analysis of Cements, Fire-clays, &c.

1. *Moisture.*—Heat about 5 gms. in a platinum crucible in the steam-oven for one hour, or till loss ceases.

The loss in weight represents moisture.

2. *Organic and Volatile Matter* (CO_2).—Ignite the same sample for fifteen minutes, and then cool and weigh.

Loss in weight represents organic and volatile matter.

3. *SiO_2, Fe_2O_3 and Al_2O_3, CaO and MgO.*—Weigh about 2 gms. accurately, and mix with five times its weight of fusion mixture. Fuse over a strong burner or blowpipe until all effervescence ceases (twenty minutes).

Note.—The mixture should not more than half fill the crucible.

When the fusion is finished, let the crucible and its contents cool. Then place it in a deep evaporating dish, and boil with water to extract the fused mass. After a quarter of an hour cover the dish with a clock-glass, and add drop by drop, strong hydrochloric acid. The clock-glass must be raised each time with care, so that as little loss occurs, due to effervescence, as possible.

When the solution is completed, wash the crucible, and add the washings to the dish, then take away the clock-glass, and evaporate the contents to dryness on the water-bath. When dry, transfer to an air-oven, and heat for one hour at 150°, to render the SiO_2 insoluble.

At the end of this time add a few drops of strong HCl, and then a few c.cms. of hot distilled water. Stir well, and filter from SiO_2. Keep the filtrate and the washings. The

SiO_2 must be collected, and ignited in a crucible to obtain its weight.

Fe_2O_3 and Al_2O_3.—Add to the filtrate strong AmOH in excess, and stir. Boil, and then filter off the ppt. of Fe_2O_3 and Al_2O_3.

This ppt. must be dried and ultimately ignited in a crucible. The weight gives the amount of Fe_2O_3 and Al_2O_3.

Filtrate from this ppt. is used for estimating lime and magnesia.

CaO.—If the bulk of liquid is too large at this stage, evaporate down somewhat, then add AmOH if needed, to make the solution alkaline, and then ammonium oxalate solution in slight excess. Stir well, and then allow the ppt. to subside.

When the ppt. of calcium oxalate has completely settled, pour the supernatant liquid through a filter, wash the ppt. twice by decantation, and then throw it on to the filter and wash again. Keep the filtrate.

This ppt. must be collected and dried, transferred to a crucible and ignited at a red heat if the weight does not exceed 1 gm., so that the residue ultimately consists of calcium oxide (CaO).

If the ppt. weighs more than 1 gm. it must only be ignited to $CaCO_3$ by heating in the crucible for twenty minutes, so that the bottom of the crucible is *just red when shaded from direct sunlight.*

MgO.—The filtrate from the calcium oxalate must be evaporated to dryness, and the residue ignited gently till all white fumes of AmCl cease. Cool the dish, treat the residue with a little strong hydrochloric acid, warm, then add water. Stir well, and filter if necessary.

To the filtrate which contains the magnesia add AmOH in moderate excess, and then sodium phosphate solution, in quantity sufficient to precipitate the magnesia. Let the vessel stand for five or six hours, and then filter off the precipitated $Mg(NH_4)PO_4$. Wash the ppt. on the filter with dilute AmOH, and then dry and remove to a crucible. Ignite the ppt. and weigh it as $Mg_2P_2O_7$.

4. *Alkalis (Na_2O, K_2O).*—Weigh accurately about 1·5 gms. of the substance, and mix it, in a finely powdered condition,

with 1·5 gms. of powdered AmCl, and 9 gms. of $CaCO_3$ in a platinum crucible.

Heat this mixture to bright redness for one hour, either over a good Bunsen burner or in a furnace.

After this, place the cooled crucible in a dish of water, and agitate the contents for a few minutes so that the alkaline chlorides will be dissolved out entirely, together with some lime.

Filter, and mix the filtrate with AmOH and Am_2CO_3, followed by a small amount of ammonium oxalate. These reagents will precipitate the lime present. Filter, acidify the filtrate with HCl, and evaporate it to dryness on a small weighed dish.

Ignite the residue gently, and continue gentle ignition until the weight of the mixed NaCl and KCl remains constant.

If y = weight of NaCl, a = total weight of (NaCl + KCl), and p = weight of chlorine in the residue, then

$$y = \frac{p - 0{\cdot}476a.}{0{\cdot}136.}$$

The chlorine can be estimated by dissolving the mixed chlorides in distilled water, making up to a known volume, and titrating a measured quantity with standard silver nitrate.

The NaCl and KCl can be calculated to Na_2O and K_2O if the results are multiplied by 0·534 and 0·635 respectively.

It is generally held that if a Portland cement on analysis gives MgO greater than 5 per cent., or H_2CO_3 and H_2SO_4 each greater than 2·5 per cent., the samples may be rejected without physical tests, as their quality will prove inferior.

*To Estimate the Porosity of Brick.

Weigh the sample brick accurately, and place it immediately in water at ordinary temperature. At first it should be only half immersed, and then, after twenty-four hours, totally covered, and so left for another twenty-four hours.

The brick should at the end of this time be taken out of the water, dried with a cloth, and weighed.

The increase in weight gives water absorbed.

Analysis of Alloys (Brass, Bronze, Gun-metal, Solder).

The analysis of any one of these alloys may involve the separation and estimation of tin, copper, lead, iron, and zinc. A qualitative analysis of the sample should first be conducted to determine what metals are present, and the quantitative estimation is then carried out as follows :—

Dissolve between 2 and 3 gms. of the alloy (in fine turnings) in 20 c.cms. of strong HNO_3, in a small dish or beaker.

Add the acid gradually, and cover the vessel, after each addition of acid, with a clock-glass or funnel. When all the acid has been added, and vigorous action has ceased, remove the covering and digest the liquid on the water-bath for half an hour. Next, add 50 c.cms. of warm distilled water, and filter the hot liquid, collecting the white hydrated SnO_2, and wash the ppt. with hot distilled water, adding the washings to the original filtrate.

Dry the SnO_2, transfer it to a crucible, and after ignition, weigh it. Calculate the percentage of tin from the weight of SnO_2.

Add to the filtrate in a dish 20 c.cms. of dilute H_2SO_4, and evaporate down until white fumes of H_2SO_4 are evolved. Then add 50 c.cms. of distilled water (carefully), and filter off the ppt. of $PbSO_4$. Wash the ppt. twice with distilled water and once with alcohol. Keep the washings and filtrate.

The white ppt. of $PbSO_4$ is dried, transferred to a crucible, and, after ignition, weighed. From its weight the percentage of lead can be calculated.

Note.—If lead is not present, the above process may of course be omitted.

Filtrate from $PbSO_4$ may contain copper, iron, and zinc.

Pass H_2S into the liquid till saturated, and filter off the precipitated CuS. During filtration keep the funnel covered with a watch-glass, and wash the ppt. with water containing H_2S.

The CuS ppt. may be treated according to either A or B.

Method A (quicker method).—The copper sulphide is dried in the hot oven and then transferred to a Rose's crucible

(see Fig. 58). Burn the paper separately, and then add the ash to the crucible contents. Cover the sulphide with a thin layer of pure sulphur, put on the lid, and connect the gas tube to a hydrogen flask. A sulphuric acid drying-bottle should be placed between the generator and the crucible.

Let a slow stream of hydrogen pass over the contents, and gradually raise the temperature to red heat, and finally for ten minutes to a bright red. Then let the crucible cool while a current of hydrogen passes, and finally finish cooling in the desiccator.

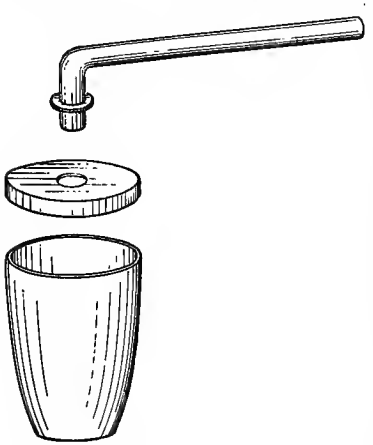

FIG. 58.—(*From Rhead's "Assaying."*)

Weigh the ppt. of cuprous sulphide (Cu_2S).

Method B. — Dissolve the CuS in a little strong HNO_3, to which a little bromine is subsequently added. Evaporate the liquid nearly to dryness, after adding a few drops of concentrated H_2SO_4.

Cool, add 50 c.cms. of water, and then precipitate the copper as CuO by boiling the solution and adding caustic potash solution till alkaline.

Filter and wash the ppt., and after drying, transfer it to a crucible, ignite, and weigh as CuO.

The filtrate from the CuS may contain Fe and Zn.

Boil off all H_2S, then add AmCl and then AmOH till alkaline. Boil and filter from $Fe(OH)_3$. Dry the precipitated $Fe(OH)_3$, and after ignition, weigh as Fe_2O_3.

The filtrate contains zinc, and must be saturated with H_2S. The white precipitate of ZnS is filtered off, washed with H_2S water, and then dissolved in dilute sulphuric acid. From this solution the zinc is precipitated by adding Na_2CO_3 in excess.

Filter from the $ZnCO_3$, transfer the dried ppt. to a crucible,

and ignite to ZnO. The filter-paper, before burning in a platinum wire, must be soaked in ammonium nitrate solution, to ensure that the ZnO on the paper shall not be reduced during burning to Zn.

The Preparation of Reagents.

Magnesia Mixture.—Dissolve 50 gms. $MgCl_2$ in 250 c.cms. of water, and mix this with 70 gms. AmCl dissolved in 200 c.cms. of water, then add 300 c.cms. of strong AmOH, and make the volume up to 1 litre. Filter the solution after standing a day or two, and label the clear solution *magnesia mixture.*

Soap Solution.—Rub together in a mortar, three parts of lead plaster (Plumbi Emplast., B.P.) with one part of potassium carbonate, in small quantities at a time. When these have been thoroughly mixed, add methylated spirit, and rub well until a creamy mass is obtained. After standing for some hours, pour the clear solution through a filter. This strong solution must be diluted by adding a mixture of equal volumes of methylated spirit and water.

Some of this is now placed in a burette, and run in, 1 c.cm. at a time to 50 c.cms. of standard calcium chloride solution, made as described on p. 147. When a lather begins to form, run the soap solution in more slowly, till finally $\frac{1}{5}$th c.cm. gives a lather which lasts for three minutes.

Dilute the soap solution further, so that 14·25 c.cms. are required to give a lather with 50 c.cms. of $CaCl_2$ solution.

Methyl Orange.—Dissolve 1 gm. of methyl orange in 200 c.cms. of methylated spirit, and make the volume up to 1 litre by adding distilled water.

Phenolphthalein.—Dissolve 5 gms. of phenolphthalein in 100 c.cms. of warm methylated spirit, and dilute to 1 litre by addition of a mixture of equal volumes of methylated spirit and water.

Platinised Asbestos.—Soak some asbestos fibre in platinum chloride solution, in an evaporating dish, using just sufficient of the liquid to thoroughly wet the fibre throughout.

Then dry the contents of the dish over a small flame, and finally ignite strongly. The asbestos will be covered with

finely divided platinum, produced by the decomposition of the chloride.

Potash for CO_2 Absorption.—Dissolve 160 gms. of potassium hydroxide in 140 c.cms. of water. This will give about 200 c.cms. of solution.

Cuprous Chloride Solution for CO Absorption.—Dissolve 27 gms. of cuprous chloride in 200 c.cms. of hydrochloric acid of specific gravity 1·124.

Alkaline Pyrogallate for Oxygen Absorption.—Dissolve 10 gms. of pyrogallic acid in 200 c.cms. of potash solution, prepared as above.

Litmus Solution.—Digest 5 or 6 gms. of coarsely powdered litmus with 200 c.cms. of distilled water, for a few hours.

Decant the clear solution from sediment and add dilute HNO_3, drop by drop, with stirring, till a violet tint is obtained.

APPENDIX

In the Tables I. and II. is given a list of the *Elements*, together with their characteristic physical properties and chemical symbols.

They are important to the Engineer—

1. As elements, Zn, Fe, Cu, Al, S, P.

2. In combination together, *e.g.* Iron oxide, Fe_2O_3; Lead sulphide, PbS; Lime, $Ca(OH)_2$; Washing soda (Sodium carbonate), Na_2CO_3, $10H_2O$.

3. Mixed with each other, in varying quantities, more particularly in alloys and amalgams, *e.g.* Brass (Cu + Zn); Type metal (Sb + Sn).

Amalgams are solutions of the metals in liquid mercury, *e.g.* Tin amalgam, Copper amalgam, &c.

The elements are classed in two divisions—

I. Metals. II. Non-metals.

and in each case in alphabetical order.

TABLE I.—

	Symbol.	Atomic Weight.	Specific Gravity.	Melting-point.	Boiling-point.	Colour.	Hardness.
Aluminium	Al	27·1	2·60	654·5° C.	...	Tin-white	2·9
Antimony.	Sb	120·2	6·62	629·5° C.	1300° C.?	Silver-white	3·0
Bismuth .	Bi	208·5	9·80	270° C.	1435° C.	Grey-white, brittle	2·5
Chromium	Cr	52·1	6·90	1515° C.	...	Iron-white	9·0
Cobalt . .	Co	59·0	8·70	1530° C.	...	Silver-white	4·5?
Copper. .	Cu	63·6	8·95	1080·5° C.	2100° C.	Red	3·0
Iron. . .	Fe	55·9	7·86	1503° C.	...	Silver-white	4·5
Lead . .	Pb	206·9	11·38	328° C.	1500° C.	Bluish-white	1·5
Magnesium	Mg	24·4	1·74	632·6° C.	1100° C.	White	2·0
Manganese	Mn	55·0	7·40	1245° C.	...	Grey	5·0
Mercury .	Hg	200·0	13·55	−38·8° C.	357° C.	Silver-white	...
Nickel . .	Ni	58·7	8·80	1427° C.	...	Silver-white	4·5
Platinum .	Pt	194·8	21·5	1710° C.	...	Tin-white	4·3
Silver . .	Ag	107·9	10·42 to 10·53	961·5° C.	1360° C. High vacuum	White	2·7
Tin . . .	Sn	119·0	7·29	232° C.	1500° C.?	White	1·8
Tungsten .	W	184·0	19·10	1500° C.?	...	Bluish-white	...
Zinc. . .	Zn	65·4	6·90	419° C.	918° C.	Bluish-white	2·5

Metals.

Conductivity.		Cubical Expansion Coefficient.	Specific Heat.	Latent Heat.		Heat of Combustion in Calories.
Heat.	Electricity.			Fusion.	Vaporisation.	
0·3619	$32·4 \times 10^4$	0·000069	0·2189	...	...	Al_2O_3, 380,200
0·042	$2·47 \times 10^4$	0·000033	0·0495	...	...	Sb_2O_5, $3H_2O$, 228,000
0·0164	$0·624 \times 10^4$	0·000039	0·0304	12·6	...	Bi_2O_3, 19,900
...	...	...	0·1208	...	...	...
...	$8·32 \times 10^4$	0·000036	0·1030	...	...	CoO, 63,800
0·7226	$55·4 \times 10^4$	0·000051	0·0936	...	...	CuO, 37,200
0·1528	$10·37 \times 10^4$	0·000037	0·1162	...	...	Fe_2O_3, 195,000
0·0764	$5·18 \times 10^4$	0·000084	0·0310	5·4	...	PbO, 50,300
0·3760	$24·5 \times 10^4$	0·000081	0·2460	...	...	MgO, 143,300
...	...	...	0·1217	...	...	MnO, 90,800
0·0177	$1·06 \times 10^4$	0·000182	0·0319	2·8	62·0	HgO, 30,600
0·1384	$9·73 \times 10^4$	0·000039	0·1084	4·6	...	NiO, 59,700
0·1861	$8·56 \times 10^4$	0·000027	0·0323	27·2	...	$Pt(OH)_2$, 17,900
1·096	$60·8 \times 10^4$	0·000057	0·0562	21·1	...	Ag_2O, 5,900
0·1446	$9·61 \times 10^4$	0·000069	0·0551	14·3	...	SnO, 70,300
...	...	...	0·0336	...	...	...
0·303	$17·43 \times 10^4$	0·00009	0·0935	28·1	...	ZnO, 85,000

TABLE II.—

	Symbol.	Atomic Weight.	Specific Gravity.	Melting-point.	Boiling-point.	Colour.
Arsenic . .	As	75·0	5·73	500° C.	...	Steel-grey crystalline
Boron . .	B	11·0	2·45	Electric furnace	Volatile in electric furnace	Brown-yellow crystalline
Bromine .	Br	80·0	3·14	−7·3° C.	59·3° C.	Deep-red liquid
Carbon . .	C	12·0	Amorphous, 1·57 Graphite, 2·5 Diamond, 3·5	...	...	Black crystalline or amorphous
Chlorine .	Cl	35·5	Boiling-point 1·56	−102° C.	−33·6° C.	Green-yellow, gaseous
Fluorine .	F	19·0	Boiling-point 1·11	−223° C.	−187° C.	Pale-yellow, gaseous
Hydrogen .	H	1·008	Boiling-point 0·07	−258·9° C.	−252·5° C.	Colourless gas
Iodine . .	I	127·0	4·95	114·2° C.	184·3° C.	Black-grey crystals
Nitrogen .	N	14·0	Boiling-point 0·81	−210·5° C.	−195·5° C.	Colourless gas and liquid
Oxygen . .	O	16·0	Boiling-point 1·12	−223° C.	−182·5° C.	Colourless gas, pale-blue liquid
Phosphorus	P	31·0	Yellow, 1·83 Red, 2·11	44·3° C.	287·3° C.	Pale-yellow crystals, red amorphous
Silicon . .	Si	28·4	2·35 to 2·49	Electric furnace	Electric furnace	Black crystals
Sulphur .	S	32·0	1·96 to 2·07	119° C.	445° C.	Yellow crystals

Non-Metals.

Hardness.	Conductivity.		Cubical Expansion Coefficient.	Specific Heat.	Latent Heat.		Heat of Combustion in Calories.
	Heat.	Electricity.			Fusion.	Vaporisation.	
3·5	...	...	0·000018	0·0830		...	As_2O_3, 154,000
9·5	...	$0·125 \times 10^{-6}$	...	0·3066	...	...	B_2O_3, 272,600
...	...	...	...	0·0843	16·2	...	...
10·0 Diamond	0·0117	$0·087 \times 10^{4}$	0·000016	0·190 to 0·204	...	...	CO, 29,000 CO_2, 97,600
...	...	...	0·0019 Liquid	0·226 Liquid	...	...	Cl_2O, −17,600
...	...	...	...	...	...	...	...
...	$0·0_3327$ Gas	...	0·00367 Gas	c.p. 3·409 Gas	16·0	200	H_2O, 68,370
...	...	...	0·00025	0·0541	...	...	I_2O_5, 45,000
...	$0·0_3052$ Gas	...	0·00367	c.p. 0·2438 Gas	...	...	N_2O_3, −21,000
...	$0·0_3056$ Gas	...	0·00367	c.p. 0·2175 Gas	...	56	...
0·5	...	$0·957 \times 10^{-11}$	0·00037	0·202	5·2	...	P_2O_5, 369,000
7·0	...	...	0·000023	0·1730	...	...	...
2·0	0·00063	$0·254 \times 10^{-15}$	0·00022	0·1844	9·4	363	SO_2, 69,260 SO_3, 91,900

TABLE III.—*Common Minerals.*

Mineral.	Substances Present.	Composition.
Alum stone . .	Potassium sulphate and aluminium sulphate	$K_2Al_2(SO_4)_4$, xH_2O.
Anhydrite . .	Calcium sulphate	$CaSO_4$.
Antimony ochre	Antimony oxide	Sb_2O_4.
Apatite . . .	Phosphate and chloride of lime	$3Ca_3(PO_4)_2$, $CaCl_2$.
Arsenite . . .	Arsenic oxide	As_4O_6.
Arsenical iron .	Iron arsenide	$FeAs_2$.
Asbestos . . .	Silicate of magnesia	$3MgO$, Fe_2O_3, $3SiO_2$.
Azurite . . .	Carbonate and hydroxide of copper	$2CuCO_3$, $Cu(OH)_2$.
Barytes . . .	Sulphate of baryta	$BaSO_4$.
Bauxite . . .	Aluminium oxide and iron oxide	$(AlFe)_2O_3$, $2H_2O$.
Borax	Sodium borate	$Na_2B_4O_7$, $10H_2O$.
Bismuthite . .	Bismuth sulphide	Bi_2S_3.
Calamine . . .	Zinc carbonate	$ZnCO_3$.
Calcite	Calcium carbonate	$CaCO_3$.
Carnallite . .	Chloride of potassium and magnesium	KCl, $MgCl_2$, $6H_2O$.
Cinnabar . . .	Sulphide of mercury	HgS.
China clay . .	Silicate of aluminium	Al_2O_3, $2SiO_2$, $2H_2O$.
Chrome iron ore	Oxide of chromium and iron	FeO, Cr_2O_3.
Copper glance .	Sulphide of copper	Cu_2S.
Copper pyrites .	Sulphide of copper and iron	$(CuFe)S_2$.
Corundum . .	Oxide of aluminium	Al_2O_3.
Cryolite . . .	Fluoride of aluminium and sodium	AlF_3, $3NaF$.
Dolomite . . .	Magnesium and calcium carbonate	$(CaMg)(CO_3)_2$.
Franklinite . .	Oxide of zinc, iron, and manganese	$(ZnFeMn)O$ + $(FeMn)_2O_3$.
Fluor spar . .	Fluoride of calcium	CaF_2.
Galena	Sulphide of lead	PbS.
Garnet	Silicate of lime and alumina	$Ca_3Al_2(SiO_4)_3$.
Gypsum . . .	Sulphate of calcium	$CaSO_4$, $2H_2O$.

TABLE III.—*Common Minerals (continued).*

Mineral.	Substances Present.	Composition.
Hæmatite . . .	Oxide of iron	$2Fe_2O_3$, $3H_2O$.
Ilmenite . . .	Oxide of iron and titanium	$(FeTi)_2O_3$.
Kainite . .	Potassium sulphate and magnesium sulphate	K_2SO_4,$MgSO_4$,$MgCl_2$, $5H_2O$.
Kupfer nickel .	Nickel arsenide	NiAs.
Magnesite. . .	Carbonate of magnesia	$MgCO_3$.
Magnetite . .	Oxide of iron	Fe_3O_4.
Malachite . . .	Basic carbonate of copper	$CuCO_3$, $Cu(OH)$.
Monazite . .	Oxide of thoria, &c.	...
Nitre	Nitrate of potassium	KNO_3.
Orpiment . . .	Sulphide of arsenic	As_2S_3.
Phosphorite . .	Phosphate of lime	$Ca_3(PO_4)_2$.
Pitchblende . .	40–90 per cent. uranium oxide	U_3O_8.
Pyrites. . . .	Iron sulphide	FeS_2.
Pyrolusite. . .	Oxide of manganese	MnO_2.
Quartz	Silica	SiO_2.
Rock salt . . .	Sodium chloride	NaCl.
Saltpetre . . .	Nitrate of soda	$NaNO_3$.
Spathic iron ore	Carbonate of iron	$FeCO_3$.
Stibnite . . .	Sulphide of antimony	Sb_2S_3.
Sylvine . . .	Chloride of potassium	KCl.
Tin-stone . . .	Oxide of tin	SnO_2.
Witherite . . .	Carbonate of baryta	$BaCO_3$.
Wolfram . . .	Tungstate of iron and manganese	$(FeMn)WO_4$.
Zinc blende . .	Sulphide of zinc	ZnS.

TABLE IV.—*List of Formulæ with Chemical Names.*

AmOH or NH_4OH	Ammonium hydroxide or hydrate.
AmCl or NH_4Cl	,, chloride.
Am_2CO_3 or $(NH_4)_2CO_3$	,, carbonate.
$Am_2C_2O_4$ or $(NH_4)_2C_2O_4$	,, oxalate.
Am_2S or $(NH_4)_2S$	,, sulphide.
AmAc	,, acetate.
$Al_2(SO_4)_3$	Aluminium sulphate.
$Al(OH)_3$	,, hydroxide.
Al_2O_3	,, oxide. (Alumina.)
As_2O_3	Arsenic oxide or white arsenic.
As_2S_3	,, sulphide.
$AgNO_3$	Silver nitrate.
AgCl	,, chloride.
$BaCl_2$	Barium chloride.
$Ba(OH)_2$	,, hydroxide or bartya.
$BaSO_4$	,, sulphate.
$BaCrO_4$	,, chromate.
$Bi(NO_3)_3$	Bismuth nitrate.
$BiCl_3$	,, chloride.
Bi_2S_3	,, sulphide.
Bi_2O_3	,, oxide.
$CdSO_4$	Cadmium sulphate.
CdO	,, oxide.
CdS	,, sulphide.
Ca(OH)	Calcium hydroxide or hydrate.
$CaCl_2$	,, chloride.
$CaSO_4$	,, sulphate.
$CaCO_3$	,, carbonate.
CaC_2	,, carbide.
$Ca_3(PO_4)_2$	,, phosphate.
CaC_2O_4	,, oxalate.
$Cr_2(SO_4)_3$	Chromium sulphate.
Cr_2O_3	,, oxide.
$Co(NO_3)_2$	Cobalt nitrate.
$CuSO_4$	Copper sulphate.
$Cu(OH)_2$	,, hydroxide.
$CuCO_3$	,, carbonate.
CS_2	Carbon bisulphide.

TABLE IV.—*List of Formulæ with Chemical Names (continued).*

Fe_2O_3	Ferric oxide or iron oxide.
$FeSO_4$	Ferrous sulphate.
$FeCl_3$	Ferric chloride.
FeS	Ferrous sulphide.
$Fe(OH)_3$	Ferric hydroxide.
HCl	Hydrochloric acid or hydrogen chloride.
H_2SO_4	Sulphuric acid or hydrogen sulphate.
HNO_3	Nitric acid or hydrogen nitrate.
HAc or $HC_2H_3O_2$	Acetic acid or hydrogen acetate.
H_2S	Hydrogen sulphide.
HgO	Mercuric oxide.
$HgCl_2$	,, chloride.
HgS	,, sulphide.
K_4FeCy_6	Potassium ferrocyanide.
KCl	,, chloride.
$KClO_3$	,, chlorate.
KNO_3	,, nitrate.
K_2CO_3	,, carbonate.
KOH	,, hydroxide.
K_2SO_4	,, sulphate.
K_2SO_3	,, sulphite.

Note—These with Na in place of K, give corresponding formulæ of sodium salts.

$MnSO_4$	Manganese sulphate.
MnS	,, sulphide.
$MgSO_4$	Magnesium sulphate.
$MgCO_3$	,, carbonate.
$Mg(OH)_2$	,, hydroxide.
$MgAmPO_4$	,, ammonium phosphate.
$NiSO_4$	Nickel sulphate.
$Ni(OH)_2$	,, hydroxide.
NiS	,, sulphide.

TABLE IV.—*List of Formulæ with Chemical Names (continued).*

$PbSO_4$	Lead sulphate.
PbS	„ sulphide.
$PbAc$	„ acetate.
$Pb(NO_3)_2$	„ nitrate.
$PbCl_2$	„ chloride.
$PbCrO_4$	„ chromate.
Pb_3O_4	„ oxide. (Red lead.)
PbO	„ oxide. (Litharge.)
$SrSO_4$	Strontium sulphate.
$SrCO_3$	„ carbonate.
$SrCl_2$	„ chloride.
SnO_2	Stannic oxide.
$SnCl_2$	Stannous chloride.
$SnCl_4$	Stannic chloride.
SnS	Stannous sulphide.
WO_3	Tungstic oxide.
ZnO	Zinc oxide.
ZnS	„ sulphide.
$ZnSO_4$	„ sulphate.
$Zn(OH)_2$	„ hydroxide.
$ZnCrO_4$	„ chromate.

TABLE V.—*Naturally Occurring Forms of Silica and Silicates.*

Silica occurs in the following forms:—

Quartz or rock crystal: clear, colourless, and transparent. This is the Brazilian pebble of spectacle makers.

Amethyst: yellow to purple, contains traces of iron and manganese.

Milky quartz. Rose quartz.

Jasper: opaque, red, brown, yellow, or green.

Hornstone. Flint. Chalcedony. Agate. Opal.

Silicates.	Bases Present.	Composition.
Asbestos . . .	Magnesium oxide Calcium oxide Iron or aluminium oxides	$3MgO, Fe_2O_3, 3SiO_2$.
Jade . . .	Magnesium oxide Calcium oxide	...
Meerschaum . Serpentine .	A hydrated silicate of magnesium	...
Topaz . . .	A silicate and fluoride of aluminium	$2Al_2O_3, 3SiO_2, 2AlF_3$.
Garnet	Oxide of calcium, aluminium, iron, and alkalis	$3CaO, Al_2O_3, 3SiO_2$.
Mica	Oxides of aluminium and potassium	$2Al_2O_3, 3SiO_2$.
Hornblende . .	Oxides of sodium and iron	Na_2O, FeO, Fe_2O_3, $5SiO_2$.
Felspar . . .	Oxides of aluminium and potassium	$Al_2O_3, 6SiO_2, K_2O$.
Beryl	Oxides of beryllium and aluminium	$3BeO, Al_2O_3, 6SiO_2$.
Kaolin or china clay	Oxide of aluminium	$Al_2O_3, 2SiO_2, 2H_2O$.
Tourmaline . .	Silicate of magnesia and lime	$(MgCa)SiO_3$.

TABLE VI.—*Specific Gravity, &c., of Some Non-Metallic Materials.*

Substance.	Specific Gravity.	Weight of 1 Cubic Foot in lbs.	Number of Cubic Feet in 1 Ton.
Brickwork (common)	1·6 to 2·0	100 to 125	22·5 to 18·0
,, (red) . .	2·16	134	16·8
Cement	1·7	106	21·1
Chalk (lumps) . .	2·0	125	17·9
,, (powder) . .	2·64	165	13·6
Clay	1·9	119	18·8
Concrete	1·9	119	18·8
Fireclay	2·52	157	14·2
Granite	2·64	164	13·6
Limestone	2·52	157	14·1
Masonry	2·1	131	17·1
Mortar	1·6	102	22·5
Quartz	2·55 to 2·77	159 to 173	14·1 to 12·9
Sand (coarse) . . .	1·61	100	22·4
,, (fine)	1·52	95	23·6
Sandstone	2·5	156	14·3
Slate	2·7	163	15·1

TABLE VII.—*Freezing Mixtures.*

Ingredients.	Proportional Parts.	Lowering of Temperature.
Sodium nitrate and water	75 parts to 100 water	18° C.
Calcium chloride (crystals) and H_2O . .	25 parts to 10 water	22° C.
Ammonium nitrate and water	60 parts to 100 water	26° C.
Sodium chloride and snow	33 parts to 100 snow	30° C.
Calcium chloride (crystals) and snow . .	100 parts to 70 snow	60° C.

TABLE VIII.—*Composition of Cements.*

Portland Cements.

SiO_2.	Al_2O_3.	Fe_2O_3.	CaO.	MgO.	Na_2O.	SO_3.	K_2O.	CO_2.
24·2	6·22	3·00	62·67	1·22	1·96	0·67	...	...
21·7	6·82	2·37	62·26	1·48	0·98	1·20	1·84	1·3
19·05	7·90	5·48	63·62	1·87	0·36	0·94	0·78	...

Natural Cements.

SiO_2.	Al_2O_3 & Fe_2O_3.	CaO.	MgO.	CO_2.	Alkali & Loss.
23·0	17·0	36·0	16·0	5·0	3·0
21·0	7·0	44·0	7·0	11·0	10·0

Val de Travers' Rock Asphalt.

Bituminous Matter.		Mineral Matter.
Petrolene.	Asphaltene.	
Per Cent. 8·5	Per Cent. 3·9	87·56

Puzzuolana or Volcanic Tuffa from Rome.

SiO_2.	Al_2O_5 & Fe_2O_3.	CaO.	MgO.	H_2O & CO_2.	Alkalis.
53·0	29 8	5·68	0·35	4·7	6·72

TABLE IX.—*Scale of Hardness (Mohr's).*

Talc = 1. Gypsum = 2. Calc-spar = 3. Fluor-spar = 4.
Apatite = 5. Orthoclase = 6. Quartz = 7. Topaz = 8.
Corundum = 9. Diamond = 10.

TABLE X.—*Action on Metals and Alloys of Dilute Salts at ordinary*

Metals and Alloys.	Sulphuric Acid.	Hydro-chloric Acid.	Nitric Acid.	Acetic Acid.	Carbonic Acid.
Aluminium . .	Weak	Weak	Weak	Weak	No action
Bell metal . . .	Weak	Very strong	Weak	Weak	No action
Bismuth . . .	Fairly strong	Fairly strong	Fairly strong	Fairly strong	No action
Brass	Fairly strong	Fairly strong	Fairly strong	Weak	No action
Copper	Weak	Fairly strong	Weak	Weak	Very weak
Ferro-manganese	Strong	Strong	Strong	Strong	Weak
German silver .	Fairly strong	Fairly strong	Fairly strong	Weak	No action
Iron, cast . . .	Strong	Strong	Fairly strong	Fairly strong	Weak
Iron, wrought .	Very strong	Strong	Strong	Strong	Fairly strong
Lead	No action	Weak	Strong	Fairly strong	Weak
Magnesium . .	Very strong	Very strong	Fairly strong	Strong	Weak
Solder	Fairly strong	Fairly strong	Strong	Fairly strong	No action
Tin	Weak	Weak	Fairly strong	Weak	No action
Zinc	Strong	Strong	Strong	Fairly strong	Weak

Note 1.—These results compare the solvent action of the above solutions, upon 1 *square decimetre* of surface per month.

(1 *to* 5 *per Cent.*) *Solutions of Acids, Alkalis, and Temperatures.*

Tap Water Only.	Ammonia.	Sodium Chloride.	Sodium Carbonate.	Calcium Chloride.	Magnesium Chloride.
No action	Weak	Weak	Weak	Very weak	Weak
No action	Fairly strong	Weak	No action	No action	No action
No action	No action	...	...	...	No action
No action	Fairly strong	No action	No action	No action	No action
Weak	Weak	Weak	No action	Very weak	Weak
No action	No action	Weak	Weak	Weak	Weak
No action	Weak	No action	No action	No action	No action
No action	No action	...	No action	Weak	No action
Fairly strong	No action	Weak	...	Weak	Weak
Weak	No action	Very weak	No action	Very weak	Weak
Weak	No action	Weak	Weak	Fairly strong	Strong
No action	No action	Weak	No action	No action	No action
Weak	Weak	Weak	Weak	Weak	Weak
Weak	Weak	No action	No action	Weak	Weak

Note 2.—The above terms convey the following meaning :—
Weak action signifies the solution of less than 1 gm. per month.
Fairly strong action signifies the solution of 1 to 10 gms. per month.
Strong action signifies the solution of 10 to 50 gms. per month.
Very strong action signifies the solution of more than 50 gms. per month.

TABLE XI.—*Calorific Value of Solid Fuels.*

	Carbon Content.	Specific Gravity.	Calorific Value in Calories.
	Per Cent.		
Dry coals, long flame . . .	75–80	1·25	8000–8500
Gas coals, long flame . . .	80–85	1·28	8500–8800
Caking coals	84–89	1·30	8800–9300
Caking coals, short flame . .	88–91	1·3 to 1·35	9300–9600
Anthracite	90–93	1·33 to 1·4	9000–9500
Oven coke	90	1·2 to 1·9	8000
Brown coal, lignite (Austria and Germany)	60	...	4000–6000
Black charcoal	87–92	...	8000
Air-dried wood	40–44	...	3000
Air-dried peat	30	...	3000–5000

TABLE XII.—*Calorific Value of Liquid Fuels.*

	Carbon.	Hydrogen.	Calories.
	Per Cent.	Per Cent.	
Tar	85	5	8,900
Creosote oil	83	10	9,000
Ostatki (petrol residue) . .	85	10	11,000
Shale oil	...	...	10,100
Russia (crude light oil) . .	86 (approx.)	13 (approx.)	12,560
Pennsylvania	86 (approx.)	13 (approx.)	9,963
Russia (crude heavy) . . .	86 (approx.)	13 (approx.)	10,800

TABLE XIII.—*Calorific Value of Gaseous Fuels.*

Coal gas	170	calories per cub. ft.
Natural gas (petroleum districts)[1]	222	,, ,,
Water-gas	74	,, ,,
Dowson-gas	36	,, ,,
Producer-gas	28	,, ,,

[1] Composition of three samples of natural gas :—

	Hydrogen.	Methane.	Ethane.	Nitrogen.
1	36·0	49·5	12·5	...
2	20·6	72·3	3·6	...
3	2·3	92·6	...	3·5

TABLE XIV.—*Proximate Analyses of Coal.*

	Moisture.	Ash.	Sulphur.	Coke.	Volatile Matter.
Non-caking	11·30	1·03	0·39	57·21	31·49
Caking	3·50	3·90	0·92	63·18	33·32
Cannel coal	1·60	3·48	0·86	63·25	35·15
Anthracite	2·00	1·61	0·91	87·20	10·80

TABLE XV.—*Products Obtained by the Fractional Distillation of Coal Tar.*

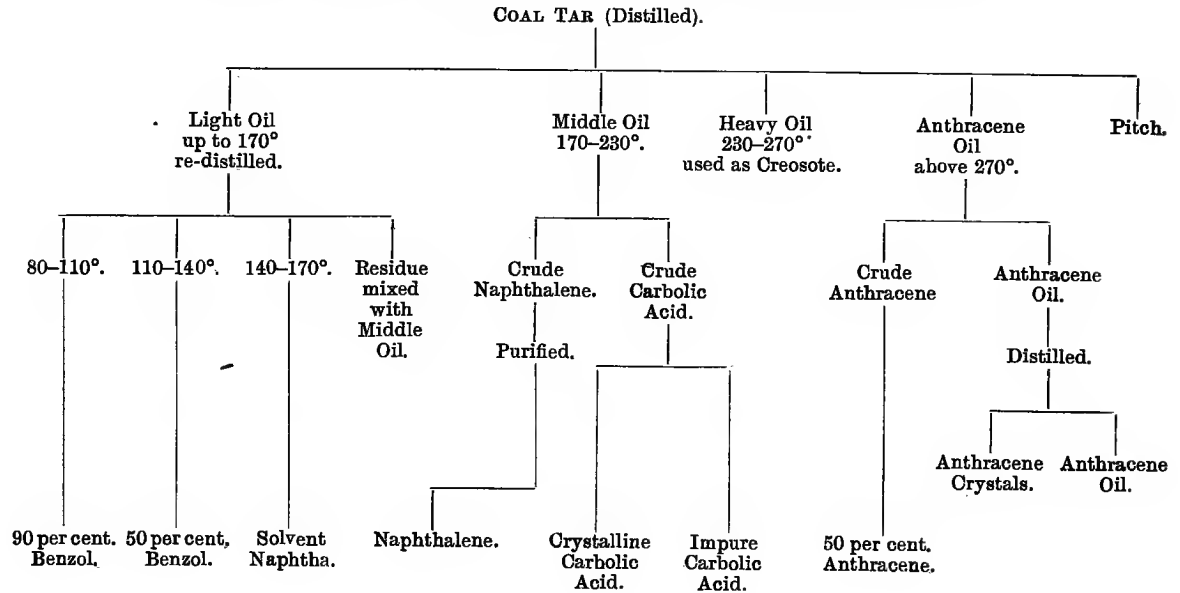

TABLE XVI.—*Typical Analyses of Furnace Gases.*

	CO_2.	CO.	H_2.	Methane.	Nitrogen.	Nature of Gas.
1	6·9	29·8	5·4	2·2	55·7	Producer-gases
2	5·2	24·4	8·6	2·4	59·4	Producer-gases
3	6·2	26·0	18·4	0·3	49·1	Dowson-gas
4	15·0	12·0	28·0	1·8	43·2	Mond-gas
5	3·6	22·8	2·7	7·4	63·5	Producer-gas

TABLE XVII.—*Flue Gases after Combustion* (*Exit Gases*).

	CO_2.	O_2.	CO.	N_2.
1	11·2	8·4	1·1	79·3
2	5·80	13·80	...	80·4
3	9·30	9·10	0·80	80·8

TABLE XVIII.—*Approximate High Temperatures.*

Just visible redness	520° C.
Dull red heat	700° C.
Cherry red heat	850° C.
Bright red heat	1000° C.
Orange-coloured heat	1100° C.
White heat	1300° C.
Dazzling white heat	1500° C.

TABLE XIX.—*Temperatures of Various Flames.*

Bunsen burner	1870° C.
Hydrogen flame	1900° C.
Coal-gas blowpipe	2200° C.
Oxyhydrogen blowpipe	2420° C.
Acetylene burner	2550° C.
Electric furnace	3000° C.

TABLE XX.—*Analyses of Various Natural Waters (Grains per Gallon).*

	$CaCO_3$.	$CaSO_4$.	$MgCl_2$.	$MgSO_4$.	NaCl.	$MgCO_3$.	
1	10·9	3·05	...	...	1·8	1·27	Thames
2	0·32	21·56	...	...	17·64	5·66	Trent
3	0·86	0·12	...	...	0·72	0·36	Scotch waters (soft)
4	2·23	0·14	...	...	1·28	1·07	
5	3·5	93·0	220·0	140·0	1855·0	traces	Sea water
6	23·76	7·33	6·28	...	29·80	...	Hard waters
7	19·60	16·10	28·74	...	20·25	...	

TABLE XXI.—*Analyses of Boiler Incrustations.*

	$CaSO_4$.	$CaCO_3$.	$Mg(OH)_2$.	Sand, Fe_2O_3, Al_2O_3.	Moisture.	Organic Matter.	NaCl.
1	85·50	1·00	3·41	1·42	5·90	Trace	2·77
2	84·27	...	7·10	2·98	5·65	...	...
3	59·21	6·07	11·19	2·85	1·36	19·32	...
4	...	62·71	4·21	9·27	2·51	3·15	...
	Magnesium carbonate = 18·15.						

TABLE XXII.—*Analyses of Cylinder Deposits.*

	Moisture.	Oils Soluble in Ether.		Fixed Carbon.	Hydrocarbons Insoluble in Ether.	FeO.	CuO.	Soap.	Al_2O_3, Fe_2O_3, SiO_2.	$CaCO_3$ and $MgCO_3$.
		Animal.	Mineral.							
1	2·28	10·54	11·23	23·73	47·97	2·83	(Undetermined = 1·42)			
2	13·12	8·15	7·86	2·71	1·67	...	...	2·10	10·46	53·39
					(Undetermined = 0·54)					
3	16·16	26·19	32·50	7·92	...	15·1	0·5	...	...	...
					(Undetermined = 1·63)					
4	3·77	21·27	19·60	10·90	...	14·01	1·07	...	...	...
					(Undetermined = 0·71)					
	Metallic iron = 27·85.				Lead oxide = 0·82.					

TABLE XXIII.—*The Percentage Composition of Some Alloys.*

Name of Alloy.	Aluminium.	Antimony.	Bismuth.	Copper.	Lead.	Nickel.	Tin.	Zinc.	Magnesium.
Aluminium bronze	5–10	...	...	95–90	...	...	...	...	...
Anti-friction metal	...	12	...	...	88	...	...	...	...
Alloy for casting	85–95	...	...	...	...	15–5	...	...	...
Bell metal	...	...	...	78	...	...	22	...	...
Brass (common)	...	...	...	66·6	...	...	...	33·3	...
,, (yellow)	...	...	...	60·0	...	...	...	40	...
Britannia metal	...	6·2	...	1·8	...	...	92	...	...
Bronze coinage	...	...	...	95	...	...	4	1	...
,, (for bearings)	...	...	...	84	...	...	13	3	...
,, (anti-friction)	...	...	...	6	...	...	14	80	...
Dutch metal	...	...	...	84·6	...	...	...	15·4	...
Electrum	...	...	...	51	...	26	...	23	...
Fusible metal	...	...	50	...	25	...	25	...	...
German silver	...	...	...	50–60	...	25–15	...	25–15	...
Gun-metal	...	...	...	90	...	...	10	...	...
Magnalium	75–80	...	...	...	...	...	...	...	25–20
Pewter	...	...	...	...	20	...	80	...	...
Solder (common)	...	...	...	...	50	...	50	...	...
,, (coarse)	...	...	...	...	66·6	...	33·3	...	...
Speculum-metal	...	...	...	66·6	...	...	33·3	...	...
Type-metal	...	25	...	...	75	...	...	...	...
,, ,,	...	25	...	...	50	...	25	...	...

TABLE XXIV.—*Relation between Metric and British Systems.*

1 inch	= 2·54 cms.	1 cm.	= 0·3937 in.
1 foot	= 30·50 cms.	1 metre	= 39·37 ins.
1 mile	= 1·609 kilometres.	1 kilometre	= 0·6213 mile.
1 cb. inch	= 16·38 c.cms.	1 litre	= 0·0352 cb. foot.
1 cb. foot	= 28·32 litres.	,,	= 0·2205 gallon.
1 gallon	= 4·536 litres.	,,	= 1·76 pints.
1 oz.	= 28·35 gms.	1 gm.	= 0·035 oz.
1 lb.	= 453·6 gms.	,,	= 15·43 grains.
1 cwt.	= 50·8 kgms.	1 kgm.	= 2·2 lbs.
1 ton	= 1016 kgms.		

Useful Constants.

1 gallon = 0·1605 cb. feet = 10 lbs. water at 62° F.
1 lb. avoirdupois = 7000 grains.
1 cb. foot water = 62·3 lbs.
1 cb. foot air at 0° C. and 1 Atmosphere = 0·0807 lb.
1 cb. foot hydrogen at 0° C. and 1 Atmosphere = 0·00557 lb.

Calculation of Gas Volumes at Normal Temperature and Pressure. N.T.P.

Normal temperature corresponds to 0° C. or 273° Absolute.
Normal pressure corresponds to 760 mm. mercury.
The gas equation is—

$$PV = \frac{P_o V_o}{273} \cdot T.$$

Hence

$$V_o = \frac{PV}{P_o T} \cdot 273.$$

P = pressure under which the volume V exists.
V_o = the volume at N.T.P.
P_o = normal pressure = 760 mm.
T = temperature on absolute scale at which the gas exists.

TABLE XXV.—*Tension of Aqueous Vapour.*

In Mm. of Mercury from 5° C. to 25° C.

° C.	Mm.	° C.	Mm.	° C.	Mm.	° C.	Mm.	° C.	Mm.	° C.	Mm.	° C.	Mm.
5·0	6·53	7·9	7·96	10·8	9·66	13·7	11·68	16·6	14·06	19·5	16·86	22·4	20·14
5·1	6·58	8·0	8·02	10·9	9·73	13·8	11·76	16·7	14·15	19·6	16·97	22·5	20·26
5·2	6·62	8·1	8·07	11·0	9·79	13·9	11·83	16·8	14·24	19·7	17·07	22·6	20·39
5·3	6·67	8·2	8·13	11·1	9·86	14·0	11·91	16·9	14·33	19·8	17·18	22·7	20·51
5·4	6·72	8·3	8·18	11·2	9·92	14·1	11·98	17·0	14·42	19·9	17·28	22·8	20·64
5·5	6·76	8·4	8·24	11·3	9·99	14·2	12·06	17·1	14·51	20·0	17·39	22·9	20·76
5·6	6·81	8·5	8·29	11·4	10·05	14·3	12·14	17·2	14·60	20·1	17·50	23·0	20·89
5·7	6·86	8·6	8·35	11·5	10·12	14·4	12·22	17·3	14·70	20·2	17·61	23·1	21·02
5·8	6·90	8·7	8·40	11·6	10·19	14·5	12·30	17·4	14·79	20·3	17·72	23·2	21·14
5·9	6·95	8·8	8·46	11·7	10·25	14·6	12·38	17·5	14·88	20·4	17·83	23·3	21·27
6·0	7·00	8·9	8·52	11·8	10·32	14·7	12·46	17·6	14·98	20·5	17·93	23·4	21·40
6·1	7·05	9·0	8·57	11·9	10·39	14·8	12·54	17·7	15·07	20·6	18·05	23·5	21·53
6·2	7·10	9·1	8·63	12·0	10·46	14·9	12·62	17·8	15·17	20·7	18·16	23·6	21·66
6·3	7·14	9·2	8·69	12·1	10·53	15·0	12·70	17·9	15·26	20·8	18·27	23·7	21·79
6·4	7·19	9·3	8·75	12·2	10·60	15·1	12·78	18·0	15·36	20·9	18·38	23·8	21·92
6·5	7·24	9·4	8·81	12·3	10·66	15·2	12·86	18·1	15·45	21·0	18·49	23·9	22·06
6·6	7·29	9·5	8·86	12·4	10·73	15·3	12·95	18·2	15·55	21·1	18·61	24·0	22·18
6·7	7·34	9·6	8·92	12·5	10·80	15·4	13·03	18·3	15·65	21·2	18·72	24·1	22·32
6·8	7·39	9·7	8·98	12·6	10·87	15·5	13·11	18·4	15·75	21·3	18·84	24·2	22·45
6·9	7·44	9·8	9·04	12·7	10·95	15·6	13·20	18·5	15·84	21·4	18·95	24·3	22·59
7·0	7·49	9·9	9·10	12·8	11·02	15·7	13·28	18·6	15·94	21·5	19·07	24·4	22·72
7·1	7·54	10·0	9·16	12·9	11·09	15·8	13·37	18·7	16·04	21·6	19·19	24·5	22·86
7·2	7·59	10·1	9·23	13·0	11·16	15·9	13·45	18·8	16·14	21·7	19·30	24·6	23·00
7·3	7·65	10·2	9·29	13·1	11·23	16·0	13·54	18·9	16·25	21·8	19·42	24·7	23·13
7·4	7·70	10·3	9·35	13·2	11·31	16·1	13·62	19·0	16·35	21·9	19·54	24·8	23·27
7·5	7·75	10·4	9·41	13·3	11·38	16·2	13·71	19·1	16·45	22·0	19·66	24·9	23·41
7·6	7·80	10·5	9·47	13·4	11·46	16·3	13·80	19·2	16·55	22·1	19·78	25·0	23·55
7·7	7·86	10·6	9·54	13·5	11·53	16·4	13·88	19·3	16·65	22·2	19·90		
7·8	7·91	10·7	9·60	13·6	11·60	16·5	13·97	19·4	16·76	22·3	20·02		

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